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A Journal of Practical Chemistry

IN ALL ITS APPLICATIONS TO

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EDITED BY
SIR WILLIAM CROOKES, F.R.S., &c.

VOLUME LXXVI.—1897.

LONDON:
PUBLISHED AT THE OFFICE, 6 & 7, CREED LANE, LUDGATE HILL, E.C.
AND SOLD BY ALL BOOKSELLERS.

MDCCCXCVII.

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{ CHEMICAL NEWS,
 { AN. 7, 1898!

1894 - 1895, Jan. 10.
Mount St. Helens

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THE CHEMICAL NEWS AND JOURNAL OF PHYSICAL SCIENCE

Edited by
Wm. Crookes, F.R.S.

(WITH WHICH IS INCORPORATED THE "CHEMICAL GAZETTE")

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Friday, July 2, 1897.

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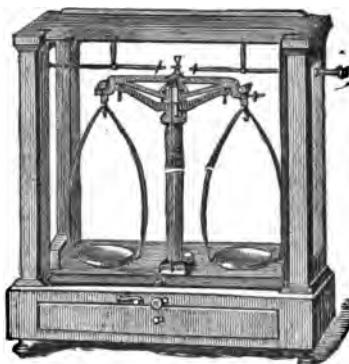


FIG. 3a.

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THE CHEMICAL NEWS.

VOLUME LXXVI.

EDITED BY WILLIAM CROOKES, F.R.S., &c.

No. 1962.—JULY 2, 1897.

DIAMONDS.*

By WILLIAM CROOKES, F.R.S., M.R.I.

(Continued from vol. LXXV., p. 302).

Depositing Floors.

OWING to the refractory character of blue ground fresh from the mines, it has to be exposed to atmospheric influences before it will pulverise under the action of water and mechanical treatment. It is brought to the surface and spread on the floors. Soon the heat of the sun and moisture produce a wonderful effect. Boulders, hard as ordinary sandstone when fresh from the mine, commence to crumble. At this stage the treatment of the diamonds assumes more the nature of farming than mining. To assist pulverisation by exposing the larger pieces to atmospheric influences, the ground is frequently harrowed and occasionally watered. The length of time necessary for crumbling the ground preparatory to washing, depends on the season of the year and the amount of rain. The longer the ground remains exposed the better it is for washing. When the process is complete the softened friable blue clay is again loaded into trucks and taken to the washing machinery, where it is agitated with water and forced through a series of revolving cylinders perforated with holes about an inch in diameter; incorrigible lumps that will not pass the cylinders are again subjected either to the weathering process or passed between crushing rollers.

Washing and Concentrating Machinery.

The fine ground which has passed through the holes in the cylinder, together with a plentiful current of water, flows into the washing pans. These pans are of iron, 14 feet in diameter, furnished with ten arms each having six or seven teeth. The teeth are set to form a spiral, so that when the arms revolve the teeth carry the heavy deposit to the outer rim of the pan, while the lighter material passes towards the centre and is carried from the pan by the flow of water. The heavy deposit contains the diamonds. It remains on the bottom of the pan and near its outer rim. This deposit is drawn off every twelve hours by means of a broad slot in the bottom of the pan. The average quantity of blue ground passed through each pan is from 400 to 450 loads in ten hours. The deposit left in each pan after putting through the above number of loads amounts to three or four loads, which go to the pulsator for further concentration.

The Pulsator.

The Pulsator is an ingeniously designed, somewhat complicated machine for dealing with the diamantiferous gravel already reduced one hundred times from the blue ground; the pulsator still further concentrating it till the stones can be picked out by hand. The value of the diamonds in a load of original blue ground is about 30s., the gravel sent to the pulsator from the pans, reduced a hundred-fold, is worth £150 a load.

The sorting room in the pulsator house is long, narrow, and well lighted. Here the rich gravel is brought in wet, a sieve full at a time, and is dumped in a heap on tables covered with iron plates. The tables at one end take the coarsest lumps, next comes the gravel which passed the 8-inch holes, then the next in order, and so on. The first sorting, where the danger of robbery is greatest, is done by thoroughly trustworthy white men. Sweeping the heap of gravel to the right, the sorter scrapes a little of it to the centre of the table by means of a flat piece of sheet zinc. With this tool he rapidly surveys the grains, seizes the diamonds, and puts them into a little tin box in front of him. The stuff is then swept off to the left, and another lot taken, and so on, till the sieve-ful of gravel is exhausted, and another brought in.

The diamond has a peculiar lustre, impossible to mistake. On the sorting table the stones look like clear pieces of gum arabic, but with an intrinsic lustre which makes a conspicuous shine among the other stones.

Watching the white men in the sorting room is an experience but tame compared to the excitement of taking a sorter's place at the big diamond table and disinterring from the gravel diamonds usually described as the finest and biggest found for many a day. The interest, however, abates when the amateur sorter is told that the jewels may not be carried away as mementos!

Sometimes as many as 8000 carats of diamonds are separated in one day, representing about £10,000 in value.

Diamonds occur in all shades, from deep yellow to pure white and jet-black, from deep brown to light cinnamon; they are also green, blue, pink, yellow, orange, and opaque.

The Diamond Office.

From the pulsator sorting room the stones are taken to the Diamond Office to be cleaned in acids and sorted into classes by the valuers, according to colour and purity. It is a sight for Aladdin to see the valuers at work in the strong-room of the De Beer's Company at Kimberley. The tables are literally heaped with stones won from the rough blue ground,—stones of all sizes, purified, flashing and of inestimable price; stones that will be coveted by men and women all the world over; and

* A Lecture delivered at the Royal Institution, Friday, June 11th, 1897.

last but not least stones that are probably destined to largely influence the development and history of a whole huge continent.

When the diamantiferous gravel has been washed down to a point at which the stones can be picked out by hand, a good plan for separating them is by their specific gravities. The following table gives the specific gravities of the minerals found on the sorting tables. I have also included the specific gravities of two useful liquids:—

	Specific gravity.
Hard graphite	2.5
Quartzite and granite	2.6
Beryl	2.7
Mica	2.8
Hornblende	3.0
METHYLENE IODIDE	3.3
DIAMOND	3.5
THALLIUM LEAD ACETATE	3.6
Garnet	3.7
Corundum	3.9
Zircon	4.4
Barytes	4.5
Chrome and titanite iron ore	4.7
Magnetite	5.0

This table shows that if I throw the whole mixture of minerals into methylene iodide, the hornblende and all above that mineral will rise to the surface; while the diamond and all minerals below will sink to the bottom. If I now take these heavy minerals, and throw them into thallium lead acetate, they will all sink except the diamond which floats and can be skimmed off.

In illustration, I have arranged an experiment. In front of the lantern is a cell containing a dense liquid; when I throw into it several minerals of different specific gravities, some sink whilst others swim, and these swimmers can easily be skimmed from the surface.

The "Compound" System.

With gems like diamonds, where infinite riches are concentrated in so small a bulk, it is not surprising that safeguards against robbery are elaborate. The Illicit Diamond Buying (I.D.B.) laws are stringent, and the searching, rendered easy by the "compounding" of the natives, is of a drastic character. In fact, it is very difficult for a native employé to steal diamonds; even were he to succeed, it would be almost impossible to dispose of them, as a potential buyer would prefer to secure the safe reward for detecting a theft rather than run the serious risk of doing convict work on the Cape Town Breakwater for a couple of years. Before the passing of the "Diamond Trade Act" the value of stolen diamonds reached nearly one million sterling per annum.

One great safeguard against robbery is the "compound" system of looking after the natives. A "compound" is a large square, about 20 acres in extent, surrounded by rows of one-storey buildings of corrugated iron. These are divided into rooms each holding about twenty natives. A high iron fence is erected around the compound, 10 feet from the buildings. Within the enclosure is a store where the necessities of life are supplied to the natives at a reduced price, and wood and water free of charge. In the middle is a large swimming-bath with fresh water running through it. The rest of the space is devoted to games, dances, concerts, and any other amusement the native mind can desire. In case of accident or illness there is a well-appointed hospital where the sick are tended. Medical supervision, nurses, and food are supplied free by the Company.

As a rule the better class of natives—the Zulus, Matabeles, Basutos, Bechuanas—when well treated, are honest and loyal.

In the compound are to be seen representatives of nearly all the picked types of African tribes. Each tribe keeps to itself, and to go round the buildings skirting the

compound is an admirable object-lesson in ethnology. At one point is a group of Zulus; next we come to Fingoes; then Basutos; beyond come Matabele, Bechuanas, Pondos, Shangains, Swasis, and other less-known tribes, each forming a distinct group, or wandering around making friendly calls. We went one afternoon to the De Beers compound when most of the natives were assembled, and having a camera with me I was naturally glad to get as many photographs as I could. I have to thank Captain Dallas, Mr. Moses, and Mr. Mandy, the Superintendents of the respective compounds, who speak all the dialects fluently, for their kindness in showing us round and improvising dances and concerts, for the benefit of my camera.

The clothing in the compound is diverse and original. Some of the men are great dandies, whilst others think that in so hot a climate a bright coloured pocket-handkerchief or "a pair of spectacles and a smile" is as great a compliance with the requirements of civilisation as can be expected.

The Diamond.

So distinctive are the characters in diamonds from each mine that an experienced buyer at once tells the locality of any particular parcel of stones. De Beers and Kimberley mines are distinguished by large yellowish crystals. Dutoitspan yields mainly coloured stones, while Bulfontein—half a mile off—produces small white stones, occasionally speckled and flawed, but rarely coloured. Diamonds from the Wesselsfontein mine are nearly all irregular in shape, a perfect crystal is rare, and most of the stones are white, few yellow. Diamonds from the Leicester mine have a frosted, etched appearance; they are white, the crystallisation irregular ("cross-grained"), and they are very hard. The newly discovered "Newlands" mines in Griqualand West are remarkable for the whiteness of their diamonds and for their many perfect octahedral crystals. Jagersfontein stones, in the Orange Free State, take the prize for purity of colour and brilliancy, and they show that so-called "steely" lustre characteristic of old Indian gems. Stones from Jagersfontein are worth nearly double those from Kimberley and De Beers.

Monster diamonds are not so uncommon as is generally supposed. Diamonds weighing over an ounce (157.5 carats) are not unfrequent at Kimberley, and there would be no difficulty in getting together a hundred of them. Not long ago, in one parcel of stones at the office of Wernher, Beit, and Co., I saw eight perfect crystals, each over an ounce and one that weighed two ounces. The largest known diamond—a true mountain of light—weighs 970 carats, over half a pound. It was found four years ago at Jagersfontein. It is perfection in colour, but has a small black spot in the centre. Diamonds smaller than a small fraction of a grain elude the sorters and are lost. A microscopic examination of blue ground from Kimberley, after treatment with appropriate solvents, shows the presence of microscopic diamonds, white, coloured, and black, also of boart and carbonado.

From two to three million carats of diamonds are turned out of the Kimberley mines in a year, and as five million carats go to the ton, this represents half a ton of diamonds. To the end of 1892, ten tons of diamonds had come from these mines, valued at £60,000,000 sterling. This mass of blazing diamonds could be accommodated in a box five feet square and six feet high.

The diamond is a luxury for which there is only a limited demand. From 4 to 4½ millions sterling is as much as is spent annually in diamonds; if production is not regulated by demand, there will be over-production, and the trade will suffer. By regulating the output, since the consolidation in 1888 the directors have succeeded in maintaining prices.

Outside companies and individuals collect diamonds to the value of about a million annually.

Graphite.

Intermediate between soft carbon and diamond come

the graphites. The name graphite is given to a variety of carbon, generally crystalline, which in an oxidising mixture of chlorate of potassium and nitric acid forms graphitic acid easy to recognise. Graphites are of varying densities, from 2.0 to 3.0, and generally of crystalline aspect. Graphite and diamond pass insensibly into one another. Hard graphite and soft diamond are near the same specific gravity. The difference appears to be one of pressure at the time of formation.

Some forms of graphite exhibit a remarkable property, by which it is possible to ascertain approximately the temperature at which graphites were formed, or to which they have subsequently been exposed. Graphites are divided into "sprouting" and "non-sprouting." When obtained by simple elevation of temperature in the arc or the electric furnace they do not sprout; but when they are formed by dissolving carbon in a metal at a high temperature and then allowing the graphite to separate out on cooling, the sprouting variety is formed. One of the best varieties is that which can be separated from platinum in ebullition in a carbon crucible. The phenomenon of sprouting is easily shown. I place a few grains in a test-tube and heat it to about 170° C., when as you see it increases enormously in bulk and fills the tube with a light form of amorphous carbon.

The resistance of a graphite to oxidising agents is greater the higher the temperature to which it has previously been exposed. Graphites which are easily attacked by a mixture of fuming nitric acid and potassium chlorate are rendered more resistant by strong heat in the electric furnace.

I will now briefly survey the chief chemical and physical characteristics of the diamond, showing you by the way a few experiments that bear upon the subject.

Combustion of the Diamond.

When heated in air or oxygen to a temperature varying from 760° to 875° C. according to its hardness, the diamond burns with production of carbonic acid. It leaves an extremely light ash, sometimes retaining the shape of the crystal, consisting of iron, lime, magnesia, silica, and titanium. In boart and carbonado the amount of ash sometimes rises to 4 per cent but in clear crystallised diamonds it is seldom higher than 0.05 per cent. By far the largest constituent of the ash is iron.

The following table shows the temperature of combustion in oxygen of different kinds of carbon:—

	°C.
Condensed vapour of carbon	650
Carbon from sugar, heated in an electrical furnace	660
Artificial graphites, generally	660
Graphite from ordinary cast-iron	670
Carbon from blue ground, of an ochrey colour	690
" " " very hard and black	710
Diamond, soft Brazilian	760
" hard Kimberley	780
Boart from Brazil	790
" from Kimberley	790
" very hard, impossible to cut	900

At the risk of repeating an experiment shown so well at this table by Professor Dewar, I will heat a diamond to a high temperature in the oxyhydrogen blowpipe and then suddenly throw it in a vessel of liquid oxygen. Notice the brilliant light of its combustion. I want you more especially to observe the white opaque deposit forming in the liquid oxygen. This deposit is solid carbonic acid produced by the combustion of the carbon. I will lead it through baryta water, and you will see a white precipitate of barium carbonate. With a little more care than is possible in a lecture I could perform this experiment quantitatively, leading the carbonic acid and oxygen, as they assume the gaseous state, through baryta-water, weighing the carbonate so formed, and showing that one gramme of diamond would yield 3.666 grammes

of carbonic acid—the theoretical proportion for pure carbon.

Some crystals of diamonds have their surfaces beautifully marked with equilateral triangles, interlaced and of varying sizes. Under the microscope these markings appear as shallow depressions sharply cut out of the surrounding surface, and these depressions were supposed by Gustav Rose to indicate the probability that the diamonds at some previous time had been exposed to incipient combustion. Rose also noted that striations appeared on the surfaces of diamonds burnt before the blowpipe. This experiment I have repeated on a clear smooth diamond, and have satisfied myself that during combustion in the field of a microscope, before the blowpipe, the surface becomes etched with markings very different in character from those naturally inscribed on crystals. The artificial striæ are cubical and closer massed, looking as if the diamond during combustion had been dissected into rectangular flakes, while the markings natural to crystals appear as if produced by the crystallising force as they were being built up.

I exhibit on a diagram a form of graphite from the Kimberley blue ground (reproduced from M. Moissan's work) which in its flaky crystalline appearance strangely resembles the surface of a diamond whose internal structure has been partially dissected and bared by combustion. It looks as if this piece of graphite was ready to separate out of its solvent as diamond, but owing to some insufficient factor it retained its graphitic form.

Physics of the Diamond.

The specific gravity of the diamond is from 3.514 to 3.518. For comparison, I give in tabular form the specific gravities of the different varieties of carbon:—

Amorphous carbon	1.45 to 1.70
Graphite	2.11 " 3.0
Hard gas coke	2.356
Boart	3.47 " 3.49
Carbonado	3.50
Diamond	3.514 " 3.518

The diamond belongs to the isometric system of crystallography. It frequently occurs with curved faces and edges. Twin crystals (macles) are not uncommon. Having no double refraction it should not act on polarised light. But as is well known, if a transparent body which does not so act is submitted to strain of an irregular character it becomes doubly refracting, and in the polariscope reveals the existence of the strain by brilliant colours arranged in a more or less defined pattern according to the state of tension in which the crystal exists. Under polarised light I have examined many hundred diamond crystals, and with few exceptions all show the presence of internal tension. On rotating the polariser, the black cross, which is most frequently seen, revolves round a particular point in the inside of the crystal, and on examining this point with a high power, we see sometimes a slight flaw, more rarely a minute cavity. The cavity is filled with gas at an enormous pressure, and the strain is set up in the stone by the effort of the gas to escape.

It is not uncommon for a diamond to explode soon after it reaches the surface, and some have been known to burst in the pockets of the miners or when held in the warm hand. Large crystals are more liable to burst than smaller pieces. Valuable stones have been destroyed in this way, and it is whispered that cunning dealers are not averse to allowing responsible clients to handle or carry in their warm pockets large crystals fresh from the mine. By way of safeguard against explosion, some dealers imbed large diamonds in raw potato to insure safe transit to England.

I will project some diamonds on the screen by means of the polarising microscope, and you will see by the colours how great is the strain to which some of them are exposed.

In the substance of many diamonds we find enclosed black uncrystallised particles of graphite. There also occur what may be considered intermediate forms between the well-crystallised diamond and graphite. These are "boart" and "carbonado." Boart is an imperfectly crystallised diamond, having no clear portions, therefore it is useless for gems. Boart is frequently found in spherical globules, and may be of all colours. It is so hard that it is used in rock drilling, and when crushed it is employed for cutting and polishing other stones. Carbonado is the Brazilian term for a still less perfectly crystallised form of carbon. It is equally hard, and occurs in porous masses, and in massive black pebbles, sometimes weighing a couple or more ounces.

Diamonds vary considerable in hardness, and even different parts of the same crystal are decidedly different in their resistance to cutting and grinding. The famous Koh-i-noor, when cut into its present form, showed a notable variation in hardness. In cutting one of the facets near a yellow flaw, the crystal became harder and harder the further it was cut into, until, after working the mill for six hours at the usual speed of 2400 revolutions a minute, little impression was made. The speed was according increased to more than 3000, when the work slowly proceeded. Other portions of the stone were found to be comparatively soft, and became harder as the outside was cut away.

Beautifully white diamonds have been found at Inverel, New South Wales, and from the rich yield of the mine and the white colour of the stones, great things were expected. A parcel of many hundred carats came to England, when it was found that they were so hard as to be practically unworkable as gems, and I believe they were ultimately sold for rock boring purposes.

I will illustrate the intense hardness of the diamond by an experiment. I place a diamond on the flattened apex of a conical block of steel, and on the diamond I bring down a second cone of steel. With the electric lantern I will project an image of the diamond and steel faces on the screen, and force them together by hydraulic power. Unless I happen to have selected a diamond with a flaw, I shall squeeze the stone right into the steel blocks without injuring it in the slightest degree.

But it is not the hardness of the diamond so much as its optical qualities that make it so highly prized. It is one of the most refracting substances in Nature, and it also has the highest reflecting properties. In the cutting of diamonds advantage is taken of these qualities. When cut as a brilliant the facets on the lower side are inclined so that light falls on them at an angle of $24^{\circ}13'$, at which angle all the incident light is totally reflected. A well-cut diamond should appear opaque by transmitted light except at a small spot in the middle where the table and culet are opposite. All the light falling on the front of the stone is reflected from the facets, and the light passing into the diamond is reflected from the interior surfaces and refracted into colours when it passes out into the air, giving rise to the lightnings and coruscations for which the diamond is supreme above all other gems.

I hold some of Mr. Streeter's magnificent diamonds in the electric light, and by transmitted light you will see they are black, while by reflected light they fill the room with radiance and colour.

The accompanying table gives the refractive indices of diamonds and other bodies. (See next column).

According to Dr. Gladstone, the specific refractive energy,—

$$\frac{\mu - 1}{d},$$

will be for the D line 0.404, and the refraction equivalent,—

$$p \frac{\mu - 1}{d},$$

will be 4.52.

After exposure for some to the sun many diamonds glow

Refractive Indices for the D Line.

Chromate of lead	2.50—2.97
Diamond	2.47—2.75
Phosphorus	2.22
Sulphur	2.12
Ruby	1.78
Thallium glass	1.75
Iceland spar	1.65
Topaz	1.61
Beryl	1.60
Emerald	1.59
Flint glass	1.58
Quartz	1.55
Canada balsam	1.53
Crown glass	1.53
Fluor-spar	1.44
Ice	1.31

in a dark room. Some diamonds are fluorescent, appearing milky in sunlight. In a vacuum, exposed to a high-tension current of electricity, diamonds phosphoresce of different colours, most South African diamonds shining with a bluish light. Diamonds from other localities emit bright blue, apricot, pale blue, red, yellowish green, orange, and pale green light. The most phosphorescent diamonds are those which are fluorescent in the sun. One beautiful green diamond in my collection, when phosphorescing in a good vacuum, gives almost as much light as a candle and you can easily read by its rays. The light is pale green, tending to white.

(To be continued).

CATHODE RAYS AND SOME ANALOGOUS RAYS.*

By SILVANUS P. THOMPSON, D.Sc., F.R.S.

1. THE size of the cathodic shadow of an object depends upon its own electric state, as already found by Crookes (*Phil. Trans.*, 1879, Part II., p. 648). If it is negatively electrified the shadow expands. If it is positively electrified the shadow contracts. The position, as well as the size of a cathodic shadow, may be affected electrostatically, the rays which cast the shadow being repelled from a neighbouring body if the latter is negatively electrified. In some cases the contraction of the shadow of a narrow object that is made positively electrical (anodic) may go so far that the luminous margins approach and even overlap, giving the appearance of a bright or negative shadow in place of a dark one. The enlargement of a shadow when the object is made cathodic, and the diminution of the shadow when the object is made anodic, both depend upon the degree of exhaustion of the tube, and both are augmented up to a certain point by raising the degree of exhaustion. They are also unequal, the enlargement when the object is made cathodic vastly surpassing the diminution when the object is made anodic, other things remaining equal. The conclusion is reached that cathode rays are capable of being deflected electrostatically, being apparently strongly repelled from a neighbouring cathodic surface, and less strongly attracted towards a neighbouring anode. Incidentally it was observed that two cathode beams from two small disc cathodes can cross through or penetrate one another without interfering with another.

2. The electrostatic deflection of cathode rays by an electrified object is found to be dependent upon the surface of that object as to whether it is a conductor or not. Objects protected by a non-conducting layer of glass do not at moderately low exhaustions, when made cathodic, repel or deflect cathode rays, and their shadow does not

* Abstract of a Paper read before the Royal Society, June 17, 1897.

enlarge. But at a certain minimum exhaustion they suddenly exert an electrostatic deflection. Naked objects made cathodic deflect the cathode rays at all exhaustions.

3. The "splash" phenomenon often observed on the tube-wall of a Crookes tube, where it is struck by the cathode beam, at the stage of exhaustion a little below that which suffices to evoke Röntgen's rays, is due to electrostatic deflections of the cathode rays by the charges on the glass.

4. A hot wire used as an object casts a cathodic shadow precisely like that of the same wire cold. Under some circumstances, if the wire is heated by an electric current, the difference between the electrostatic state of its different parts may slightly affect the size of the shadow it casts.

5. Cathode rays cannot be concentrated by reflection either from a non-conducting or a conducting surface, nor by passage through a metal tube which is itself negatively electrified.

6. When cathode rays strike upon an internal metal target or anticathode there are emitted from the latter (both at exhaustions lower than suffice to produce Röntgen rays, and at exhaustions at which those rays are also produced) some internal rays resembling ordinary cathode rays in the following respects:—They produce a similar luminescence of the glass; they cast shadows of objects; they are susceptible of deflection both magnetically and electrostatically. But they produce no Röntgen rays where they fall upon the glass surface. They do not follow either the law of specular reflection, nor that of diffuse reflection, but are emitted from the anti-cathode surface apparently according to a similarly anomalous distribution to Röntgen's rays, i.e., with nearly equal intensity, at all angles up to 90° with the normal. It is proposed to call these rays para-cathodic rays in contradistinction to the ordinary or ortho-cathodic rays. From the similarity of their distribution with that of the Röntgen rays it is inferred that the physical processes concerned in their production are identical. These para-cathodic rays are emitted from the anti-cathode both when the latter is made an anode and when it is neutral or even made cathodic. From an anti-cathode there may proceed at one and the same time, and in one and the same direction, para-cathodic rays and Röntgen rays, which, meeting an interposed object, may cast simultaneously two shadows—a para-cathodic shadow on the glass, and a Röntgen shadow on an external screen of barium platinocyanide. The former shadow can be deflected by a magnet, the latter cannot. The former shadow expands if the object is made cathodic, the latter does not.

7. If thin metal screens are used to sift the cathode rays, the luminescent phenomena change. The rays of least penetrating power appear to be most susceptible to magnetic and electrostatic forces. The various constituents of a heterogeneous cathode beam are emitted in various proportions at different degrees of exhaustion. In the cathode rays emitted at higher degrees of exhaustion there is a greater proportion of the less deflectable rays. The least deflectable rays are those which most readily penetrate through a perforated screen when that screen is itself made cathodic.

When ordinary cathode rays fall upon a perforated screen which is itself made cathodic, or are attempted to be passed through a tubular cathode, there emerge beyond the screen or tube some rays, here termed dia-cathodic rays, which differ from the ortho-cathodic and also from the para-cathodic rays. These dia-cathodic rays are not themselves directly deflected by a magnet. They show themselves as a pale blue cone or streak. Where they fall on the glass they do not excite the ordinary fluorescence of the glass. The dia-cathodic rays excite, however, a different or second kind of fluorescence, the tint in the case of soda-glass being a dark orange. Intervening objects in the beam or cone of dia-cathodic rays cast shadows. The orange fluorescence evoked on soda-glass by the dia-cathodic rays shows in the spectroscopic

the D lines of sodium only. The shadows cast by dia-cathodic rays are not deflected by the magnet, nor do they change their size when the object is electrified.

ON THE CHANGE OF ABSORPTION PRODUCED BY FLUORESCENCE.*

By JOHN BURKE, B.A. (Dub.),
Berkeley Fellow of the Owens College, Manchester.

If a body, A, of some fluorescent substance, such as uranium glass, be transmitting light from a similar body, B, which is fluorescing, the amount of light transmitted by A from B seems quite different, according as A is fluorescing or not. There appears to exist in a dilute solution of fluorescein or eosine a small difference, but a strong solution of either does not permit its fluorescent light to penetrate, except a very small thickness of the liquid, whether fluorescing or not.

Sulphate of quinine is too transparent to the luminous rays, so that even if a change did exist it could hardly be detected. The case is otherwise with uranium glass, in which the effect is well marked.

The compounds of uranium are remarkable for many characteristic properties connected with fluorescence, which they seem to exhibit more readily than most other bodies. The spectrum of the fluorescent light emitted by uranium glass shows certain maxima and minima, as was noticed by Stokes in 1852.

The experiments which have been carried out with a view to testing whether a change in the absorption is produced by fluorescence, have been almost exclusively upon uranium glass. Regarding it as the most suitable substance to experiment upon, a careful series of experiments have been made to determine whether the phenomenon really existed or not.

If we call α and β the fractions of the light from B transmitted by A, according as the latter is fluorescing or not, for uranium glass 1 c.m. thick; the values of α in a particular set of experiments, in which eye determinations were taken, were as follows. Each experiment consisted of twenty observations.

Exp. I.	..	..	..	$\alpha=0.54$
II.	..	..	..	$=0.46$
III.	..	..	..	$=0.51$
IV.	..	..	..	$=0.36$

and for β —

Exp. I.	..	..	..	$\beta=0.70$
II.	..	..	..	$=0.84$
III.	..	..	..	$=0.72$
IV.	..	..	..	$=0.92$

The mean value of α obtained from these being $\alpha=0.47$, $\beta=0.79$.

The ratio $\frac{\beta}{1+\alpha}$ was also independently determined, and found as the mean of eighty observations to be 0.507.

The values of α and β have also been determined photographically, giving—

α	<0.48	β	<0.75
	>0.43		>0.89

If we take the maximum value of α given photographically 0.48, the ratio $\frac{\alpha}{1+\alpha} = 0.32$, and instead of obtaining equality when the photometer is adjusted for this value the difference is most marked.

The effect has also been shown by obtaining two photographs on the one plate; one photograph being the result of an exposure to the light from two fluorescing cubes one behind the other, and the second photograph the result of superposing the effect of the light from A

* Abstract of a Paper read before the Royal Society, June 17, 1897

alone, when fluorescing, upon that from B after having passed through A, when the latter was not fluorescing. The exposure in each of the three cases being the same, a very distinct difference is shown in the result; the superposed photographs being always the darker in the negative, notwithstanding the fact that the resultant effect of superposing two photographs due to light of the same intensity, or nearly so, has been found not to be equal to, but less than, that due to light of double the intensity acting for half the time. If the resultant effect were equal to the sum of the separate ones, the effect caused by the change of absorption would have been still more marked.

In the determinations of α and β a null method has been employed, by which any appreciable want of uniformity in the illumination can be detected.

The source of illumination has been almost invariably the spark discharge of a Leyden jar between cadmium electrodes, being one of the richest sources of the fluorescence exciting rays, and the photometer one specially constructed for the purpose.

ON THE VOLUMETRIC DETERMINATION OF ZINC BY POTASSIUM FERROCYANIDE.*

By L. L. DE KONINCK and EUG. PROST.

I. Introduction.

THE volumetric estimation of zinc has, since the publication in 1856 (*Polytechnisches Journal de Dingler*, vol. cxi., p. 114, and vol. cxliii., p. 263; *Journal de Pharmacie*, [3], vol. xxix., p. 205, and vol. xxxi., p. 70), by Max Schaffner, of the process which now bears his name, been the subject of numerous researches, which have had for object either the improvement of this process—which at first left much to be desired—or the discovery of others.

This fact is easily accounted for by the importance of having, especially in the laboratories at mines and zinc works, a trustworthy commercial method, uniting rapidity and simplicity with a sufficient degree of exactness with regard to the value of the metal.

Of all the methods proposed, two only appear to be now in general use.

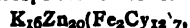
On the Continent, so far as we know, the Schaffner process is almost exclusively used; it is even the only volumetric one described, in several English and French books, among the recent methods for the analysis of zinc ores (Riche and Gelis, Silva, Halphen; Clowes and Coleman). In Germany it is certainly the most widely-used process. In America, however, it is otherwise, for, according to an interesting report made to the Scientific Society of Colorado (CHEM. NEWS, vol. lxvii., pp. 5 and 17, 1893) by a Committee charged specially to endeavour to establish uniformity among the methods used in that country, it would appear to be the Galletti process (*Bull. Soc. Chim.*, vol. ii., p. 83, 1864) modified by Fahlberg (*Zeitsch. f. Anal. Chem.*, vol. xiii., p. 379); that is to say, the process based on the precipitation of the zinc by ferrocyanide of potassium, in an acid solution, which is generally preferred in the mining districts; it is, in fact, the only one cited by the chemists who were consulted. It is also recommended by G. E. Dougherty, in a note on the Analysis of Minerals (*Engineering and Mining Journal*, 1890, p. 178; *Zeitsch. f. Angew. Ch.*, vol. iii., p. 306, 1890).

The methods of Schaffner and Galletti are the only ones which are found in the best-known treatises on

* In the CHEMICAL NEWS (vol. lxxv., p. 182) we published a short abstract of this paper; but we regret to say that the copy which came into our hands was incomplete and inaccurate. We therefore think it best to now insert the paper in full, or at any rate but slightly abridged.—Ed. C. N.

quantitative analysis of late years (Kerl, Balling, Post, Böckmann, &c.).

The sulphide of sodium process of Schaffner has been carefully studied, and thoroughly described in detail. Most writers are in accord on the details of manipulation, and notably on the use of Polka paper (salts of lead) as an indicator. This unanimity does not exist in the case of the ferrocyanide method. In this case the different writers are not even agreed as to the formula of the reaction on which the method is based. According to some the precipitate obtained should be simple ferrocyanide of zinc, $Zn_4Fe_2Cy_{12}$; according to others it should be the double ferrocyanide of zinc and potassium, $K_2Zn_2Fe_2Cy_{12}$, described long ago by Mosander (*Handwörterb. d. rein. u. Angew. Ch.*, 1848, p. 86). Others, again, maintain that the exact composition of the precipitate is more complicated still, and corresponds to the formula—



or should be even more or less indeterminate (Zulkowski, *Polyt. Journ. de Dingler*, vol. ccxlix., p. 175, 1883), for in spite of the high state of purity in which it is possible to obtain ferrocyanide of potassium, and the almost absolute stability of this reagent, they recommend the experimental determination of the relations between this salt and the quantity of zinc precipitated, this proportion not being, according to them, so simple as is generally expressed by the formulæ in common use (Mohr, Classen, 1886, p. 459).

A priori, according to the description of the process given by most writers, this would seem to be of extremely simple application, while such is not absolutely the case with the Schaffner process. Why, therefore, is the latter preferred in our continental laboratories? Is it merited, or simply a matter of custom?

It is this question that we have set ourselves to solve: one of us having, owing to a previous appointment, had a large experience in Schaffner's method, we were enabled to speak with authority when we had compared the results obtained from the careful study we made of the ferrocyanide process.

It has been proposed to apply, to the volumetric estimation of zinc, precipitation under the three following conditions:—

1. In acid solution (Galletti);
2. In simple ammoniacal solution (A. Renard);
3. In a tartaric-ammoniacal solution (Giudice).

From these spring three different processes, which we will designate by the names of their respective authors, and which we hope to study successively. We take first the Galletti process, the oldest and most widely used of the three.

II. Historical.

It was in 1864 that M. Galletti described for the first time (Mémoire presented to the Royal Academy of Science of Turin, 1864—*Bull. Soc. Chim.*, vol. ii., p. 83, 1864) the method of estimating zinc by means of ferrocyanide, a process which he had, however, foreshadowed some years previously, on the occasion of publishing an analogous process for the estimation of copper (*Ibid.*, 1856). After first separating the iron by ammonia, he added acetic acid to the filtrate, heated to 40°, then ran in the titrated solution of ferrocyanide; the end of the reaction was indicated by the sudden milky appearance of the liquid. The titration recommended is $T_{12} = 0.01$ grm. Galletti admits that the precipitate is the simple zincic ferrocyanide, and he attributes to impurities in the reagent used the fact that the results do not agree with theory, in spite of the very wide differences found. He warns one against acidulating with mineral acids, any excess interfering with the reaction, which, he says, can be perceived by the yellow colour of the liquid, due to ferrocyanide which has not been acted on. This colour does occur, but Galletti is mistaken as to its origin: we shall show later on to what this is due, and how it can be avoided. In another note published in 1868 (also *Zeitsch*

f. Anal. Chem., vol. viii., p. 135, 1869) he returns to the application of his process to the assay of minerals, with a view to modifying somewhat the method of preparing the zincic solution; but the actual estimation of zinc is done in exactly the same manner. The presence of lead has no influence; manganese is eliminated by adding bromine to the ammoniacal solution, and exposing it to the air for twenty-four hours. We shall soon come to the influence this reagent exerts. Finally, the operation can be carried on without filtering off the iron.

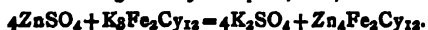
In 1874 C. Fahlberg published, as new (*Zeitsch. f. Anal. Chem.*, vol. xiii., p. 379, 1874), without referring to Galletti, the ferrocyanide process; his method of procedure was different to that of his forerunner, in that the precipitation was effected in hydrochloric solution, and, above all, by the means employed for detecting the end of the reaction. Fahlberg here utilises, by inverting it, the method used in the titration of phosphates by a titrated solution of a uranium salt,—that is to say, he uses the spot method, of a solution of uranic nitrate, to detect the brown colour which is produced the moment the potassic ferrocyanide commences to predominate. According to him, and here he is wrong, the presence of manganese would be without influence if the solution is sufficiently acid, so he does not eliminate it before titration. In each experiment the solution was acidulated with 10 to 15 c.c. of hydrochloric acid (sp. gr. 1.12 = 23.82 per cent), or 2.7 to 4 grms.; this is too much. He recommends, as does Galletti, the preparation of a solution of ferrocyanide, $T = 0.01$ grm., and the experimental determination of its strength by titrating with a solution of pure zinc dissolved in HCl; to this solution he also adds 5 parts of ammonium chloride for each 1 part of zinc, so as to obtain a precipitate "as attenuated as possible," which will subside rapidly without "carrying down potassium ferrocyanide." Fahlberg always describes the precipitate as a zincic ferrocyanide; he seems to believe, from what we have just quoted, that such is really the case.

To estimate zinc in an ore, he dissolves it in aqua regia, with excess of HCl, precipitates the copper, &c., by sulphuretted hydrogen, re-oxidises the iron salts with nitric acid, precipitates with excess of ammonia, and filters. The ammoniacal filtrate is made acid with HCl in the proportion mentioned above; the titrated solution of ferrocyanide is then run in, little by little, until the nitrate of uranium test shows the end of the operation.

Thus, as we shall show directly, the method of working described above contains sources of error which the author has quite overlooked; so it is not astonishing that the results published by him—results obtained from eleven ores from the Hartz district, containing from 3 to 23 per cent of zinc—should not be of great value; compared with those obtained dosimetrically they show errors reaching 0.49 per cent at least, on 19.77 per cent, and as much as 0.33 per cent on 3.57 per cent. This is very far from the exactness we have a right to expect, and that which can be obtained by Schaffner's method.

In the same year, 1874, Galletti published a third edition of his pamphlet (Gènes, 1874); the only modification indicated refers to the proportion of ammonia and acetic acid, to be used in a case when one wishes to titrate the zinc without previously removing the precipitated iron.

When using a zincic solution with alkaline ferrocyanides K. Zalkowski, like Fahlberg, seems to have entirely overlooked the work already done by others. When using a normal solution of ferrocyanide (105.5 grms. per litre), and a deminormal solution of a pure zincic salt, such as zincopotassic sulphate, $K_2SO_4 \cdot ZnSO_4 \cdot 6H_2O$ (mol. weight 442.57, 110.82 grm. per litre), this author readily recognises the fact that the reaction between the zinc salt and the ferrocyanide is not so simple as would appear from the formula which is generally accepted, viz.,—



(To be continued).

METHOD OF ESTIMATING ALDEHYD IN ETHER.

By M. FRANCOIS.

THE almost constant presence of ordinary aldehyd in so-called pure ethers, even that used for anæsthetic purposes, does not yet appear to have attracted sufficient attention from pharmacologists. This impurity, however, exists in a sufficiently high proportion in the products ordinarily delivered to pharmacists, and ether containing several units per cent of aldehyd is not unfrequently met with.

Thus, in medicine we admit a high proportion of impurity, which would not be tolerated in any alcoholic industry. M. Adrian (*Moniteur Scient.*, 1894, p. 835) in an excellent note, which completed the well known work of Regnault and Adrian on the estimation of ether, noted this constant presence of aldehyd, and gave a method for detecting and eliminating it. This method consisted of passing a current of dry ammonia gas through the cooled ether; crystals of aldehydate of ammonia were deposited, insoluble in ether. By this means we can detect the presence of 0.5 per cent of aldehyd. The method of purification consists of filtering and then eliminating the ammonia and rectifying.

It occurred to me to make use of the action of bisulphited rosaniline for the estimation of aldehyd in ether, and to apply to this estimation the colorimetric method of Mohler, who has rendered such great services in the analysis of alcohols. In this method (Mohler, *Moniteur Scient.*, 1890, p. 893, and 1891, p. 582) we make use of the reddish violet colouration produced by the action of bisulphited rosaniline on aldehyd. We cause this reagent to act on an alcohol, whose contents of aldehyd is known, and also on the alcohol under examination; the tints developed are observed with a Duboscq colorimeter, and by dilution we can bring the two alcohols to the same colour. From the amount of dilution we can calculate the weight of aldehyd per litre present.

To apply this method to the estimation of an ether, it is necessary to make some modifications. Mohler's reagent, which contains bisulphite of soda, precipitates sodic salts with strong alcohol and with ether; further, it will not mix with ether. We can get over this difficulty by using, for the preparation of the reagent, an aqueous solution of sulphurous acid instead of bisulphite of soda, and by adding to the ether, at the moment of making the test, its own volume of 95 per cent alcohol, free from aldehyd. With these modifications, the mixture of ether, alcohol, and reagent remains limpid, and the colorimetric determinations are easily made.

Preparation of the Reagent.—A reagent of medium sensitiveness can be obtained by using:—

Water recently saturated with SO_2	..	..	220 c.c.
Solution of fuchsine, 1/1000th	..	..	30 "
Sulphuric acid, 66°	..	..	3 "

The solutions of fuchsine and sulphurous acid are first mixed, and after agitation we add the sulphuric acid.

The reagent should be colourless. If necessary, we can filter after twenty-four hours. The fuchsine used should be the ordinary kind, not acid (Cazeneuve, *Journ. de Pharm.*, June 15, 1896). The reagent is the more sensitive as it contains less sulphuric acid. Its sensitiveness can thus become exaggerated, even to the extent of becoming coloured when mixed with absolutely pure alcohol and ether. Increasing the quantity of sulphuric acid causes this fault to disappear.

A mixture of 5 c.c. of pure ether, 5 c.c. of pure 95 per cent alcohol, and 4 c.c. of the reagent remained uncoloured for fifteen minutes. If we replace the pure ether by an ether containing more than 1/10,000th part of aldehyd, the mixture assumes a red-violet colour, the more intense as it contains more aldehyd, but not in direct proportion.

To perform the experiment it is necessary to have some freshly-made reagent, ether free from aldehyd,

solution of aldehyd in 90 per cent alcohol containing 1 grm. of aldehyd per litre, a similar solution containing 0.1 grm. of aldehyd per litre, and a Duboscq colorimeter.

Alcohol containing 1/10,000th of aldehyd will serve as a comparison for all ethers containing more than 1/10,000th of aldehyd, and alcohol at 1/10,000th for ethers containing from 1/10,000th to 1/10,000th of aldehyd.

Method of Estimation.—Into a test-tube (1) we pour 5 c.c. of alcohol containing 1/10,000th part of aldehyd and 5 c.c. of pure ether; into a second tube (2) we pour 5 c.c. of alcohol containing 1/10,000th of aldehyd and 5 c.c. of pure ether; into a third tube (3) we pour 5 c.c. of pure 95 per cent alcohol and 5 c.c. of the ether to be examined. At the same moment we add to each 4 c.c. of the reagent, shake well, cork, and note the time. After fifteen minutes observe the colour produced.

We note in the first place if the intensity of the colouration is the stronger in tube 3 than in tube 1; if it be stronger, we know that the ether contains more than 1/10,000th of aldehyd, and we therefore take tube 2 as the standard of comparison; if the colour be less strong, we know that the ether contains less than 1/10,000th of aldehyd, and we take tube 2 as the standard.*

In the case of an ether containing more than 1/10,000th of aldehyd we note the colour through a thickness of 10 m.m.

If N be the thickness of the solution under examination (tube 3), which has the same intensity of colour as the standard of comparison, the proportion x per litre of aldehyd, taking the amount present as proportional to the colour, will be given by the equation—

$$\frac{x}{1 \text{ grm.}} = \frac{10 \text{ m.m.}}{N}$$

The proportion 10/N indicates how much the ether under examination should be diluted with pure ether, so that its contents in aldehyd should be brought to 1 grm. per litre.

We now re-commence the estimation, taking tube 1 as the highest, and preparing tube 3 with ether thus diluted, and the value of N will be very near to 10 m.m. The new proportion 10/N enables us to make another dilution, more exact, and then to make a third and last trial which will give a complete uniformity of tints. From the quantity of pure ether it has been necessary to add, we deduce the quantity of aldehyd present in the ether under examination.

For an ether containing from 1/10,000th to 1/10,000th of aldehyd, we operate in the same manner, but taking tube 2 as the standard; that is to say, a solution of aldehyd of 1/10,000, and comparing the colours through a thickness of 25 m.m.

This method enables one to estimate very small quantities of aldehyd, and it can be done in less than an hour.

Preparation of Ether free from Aldehyd.—This is easily prepared by the action of permanganate of potash in alkaline solution on ether at 65°. We use anhydrous commercial ether for choice. This is placed in a stoppered litre flask with 200 c.c. of a saturated solution of permanganate of potash and 20 grms. of caustic soda. After twenty-four hours and frequent agitation, we decant by means of a funnel fitted with a tap, and submit the ether to the same treatment over again. The filtered ether is then left for twenty-four hours in contact with a mixture of 50 grms. of quicklime and 50 grms. of fused chloride of lime; it is then filtered and distilled. The permanganate has oxidised the ether and the alcohol, and the ether thus obtained gives no colouration with the aldehyd reagent, at least not after fifteen minutes.

Preparation of Alcohol free from Aldehyd.—Commercial alcohol is freed from aldehyd, furfural, and bases by adding to 1 litre of the alcohol 10 c.c. of aniline and 10

c.c. of phosphoric acid at 45° B., boiling for an hour (with a vertical condenser), and distilling.—*Journal de Pharmacie et de Chimie*, Series 6, vol. v., No. 11.

THE SMALL BESSEMER PROCESS FOR STEEL CASTINGS.

By SERGIUS KERN, M.E., St. Petersburg.

In the *CHEMICAL NEWS*, vol. lxxiii., pp. 170, 192, we published some notes on the Walrand-Légénisiel small Bessemer process. At the Baltic Shipbuilding Works, St. Petersburg, for some time past, we have used instead of the Walrand process the usual Bessemer method for making steel castings in our convertors, with a capacity of two-thirds of a ton each.

The chief reasons which induced us to discontinue the use of the *modus operandi* advised by the Walrand process are as follows:—

1. The uncertainty of obtaining uniform results. Steel castings obtained by the Walrand process may be good for the general trade, but may be variable when tested for tensile strength and elongation.

2. The extreme difficulty in obtaining steel castings which would give regular results, after being mechanically tested according to the regulations of the Russian Naval Technical Committee (28 to 35 tons per square inch of tensile strength, 14 per cent of elongation in 2 inches).

3. No necessity for the addition of ferro-silicium during the blow, in order to raise the temperature of the metal in the convertor, before the casting. In our convertors, as mentioned above, we produce, running the process by the usual Bessemer method, steel at a temperature of 2200°—2400° C., quite sufficient for our shipbuilding castings, weighing one cwt. and higher.

4. Starting with a metal from the cupola containing 4 per cent of silicon, we only lengthen the process, and make also the product obtained dearer. We start the process now, with a metal from the cupola, with 2.25 per cent of silicon, which we find quite sufficient for the charge of our convertors.

5. We certainly do not use from the very commencement, as I pointed out, Mr. Walrand's system to put the blast on, and turn the convertor up for an instant, after making the final additions, in order to mix them with the metal. Such a serious mistake will give metal with blowholes in very soft steel, or, in other cases, secondary reactions.

The following are some details of our present work while making steel castings for Government orders:—

Charges Nos. 63 and 64. (Nearly equal operations).

Charge in cupola:—

Pig-iron, Ayresome, with 4% silicon .. 900 lbs.
Runners 700 "

(This charge was melted in the cupola in 40 minutes, and run into the vessel).

Minutes from the commencement of blow.

9	D line visible; pressure 7½ lbs.
10	Green lines visible; pressure from this point to end of the operation 5½—5 lbs. per sq. inch.
18	No green lines visible; convertor turned down.

Three minutes after the appearance of the D line the convertor was turned down and the slag cleaned off. This operation took 2 minutes; time not counted in the above table.

After the disappearance of the green lines, 12 pounds of ferromanganese, containing 80 per cent of manganese, were added to the convertor, after cleaning the slags off a second time. A further charge of aluminium was made, and the steel, after the usual tests, was run into the

* It would seem to be more regular to take as standards of comparison solutions of aldehyd in pure ether; but we used solutions of pure alcohol because they last longer without changing. The results are evidently the same.

casting ladle, into which aluminium was also thrown. In all 2½ pounds of this metal was used.

The metal had a temperature of 2400° C. Prof. Wiborgh's very handy and reliable thermophone combs were used. The metal cast very quietly, and did not rise in the gates of the moulds. A casting weighing 8 cwts. was made out of the charge No. 63, and from No. 64 other castings of different weight were made.

Out of a charge of 1600 pounds in the cupola about 1200 pounds is obtained as finished steel.

The following are the chemical and mechanical analyses of our steel castings:—

	Carbon	0·11 per cent	
	Manganese ..	0·45 "	
	Silicon	0·08 "	
No. of specimen.	Tensile strength. Tons. per sq. in.	Elongation in 2 ins. P.C.	Bending test. Angle.
No. 63, annealed..	25·1	20·8	141 (a)
Ditto	26·2	15·8	
No. 64, annealed..	23·7	31·6	50 (b)
Ditto	23·5	30·1	
No. 64, unannealed	23·0	22·6	40 (c)
No. 64, forged specimens	30·3	27·8	124 (d)
Ditto	30·1	27·8	

Remarks.

(a) Bent without cracks. (b) Cracked. (c) Broke with a coarse crystalline fracture, one edge coloured brown. (d) Without cracks, there being one crack coming in forging.

In conclusion, we must state that the working out of many details and improvements gives full credit to the ingenuity of Mr. Leo Reyher, the manager of the Steel Foundry of the above-mentioned Russian Government Works.

June 14, 1897.

HONOURS FOR MEN OF SCIENCE.

THE Honours list issued on Tuesday, June 22nd, in connection with the Diamond Jubilee, contains the names of a number of men of science upon whom Her Majesty has been pleased to confer distinctions.

Dealing first with Fellows of the Royal Society, Mr. Crookes and Dr. Gowers receive knighthoods. In the Order of the Bath, Mr. Wolfe Barry, President of the Institution of Civil Engineers, Dr. Frankland, Foreign Secretary of the Royal Society, Dr. Huggins, Mr. Norman Lockyer, Director of the Solar Physics observatory, Dr. Thorne Thorne, Principal Medical Officer to the Local Government Board, and (naval promotion) Admiral Wharton, Hydrographer of the Admiralty, are appointed K.C.B.

Mr. Christie, Astronomer Royal, and Mr. Niven, Director of Studies at the Royal Naval College, are appointed C.B.

In the Order of the Star of India, Sir Joseph Hooker and Lieut.-General Strachey are promoted to the grade of G.C.S.I.

In addition to the foregoing, Baronetcies are conferred upon Sir Wm. MacCormack, President of the Royal College of Surgeons; Mr. Wilks, President of the Royal College of Physicians; and Mr. Thomas Smith, Surgeon-Extraordinary to Her Majesty. Mr. Durston, Engineer-in-Chief to the Navy, is made a K.C.B., and knighthoods are conferred upon Mr. A. R. Binnie, the Engineer to the London County Council, and Dr. Felix Semon.—*Nature*, June 24, 1897, p. 181.

Preparation of Furfurane.—P. Freundler.—Furfurane may be obtained pure and in a good proportion by heating pyromucic acid in a closed vessel to 260°—275° for two hours.—*Comptes Rendus*, cxxiv., No. 21.

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, June 25th, 1897.

Mr. SHELFORD BIDWELL, President, in the Chair.

A PAPER by Mr. SUTHERLAND, on "A New Theory of the Earth's Magnetism," was taken as read.

Dr. KUENEN described some "Experiments on Critical Phenomena," made in continuation of a research on the condensation and critical phenomena of mixtures of ethane and nitrous oxide, the results of which were published last year. The author now investigates mixtures of ethane and acetylene, and mixtures of ethane and carbonic acid, and finds for them similar properties to those of the mixtures of ethane and nitrous oxide. The first part of the paper refers to the preparation of ethane, and the effect of impurities on its vapour pressure and critical constants. Ethane from ethyl iodide is not very pure; it has generally several per cent of an admixture of a substance of higher critical temperature and higher density than ethane: this substance is probably butane. Ethane from sodic acetate, by electrolysis, is nearly pure; the method of preparation is described by the author. The pressure and corresponding volumes during condensation are given for this substance at various temperatures. In the former paper, above referred to, instances are mentioned of mixtures having critical temperatures *below* those of the component substances. The only instance of critical temperatures *above* those of the components seem to be those relating to mixtures of carbonic acid and acetylene. According to an experiment of Dewar's, however, a mixture of $\frac{1}{2}$ CO₂ and $\frac{1}{2}$ C₂H₂ has a critical temperature of 41° C., those for carbonic acid and acetylene being 31° and 37° respectively. The present investigation contradicts this result. Dewar may not have taken sufficient precaution in avoiding errors of retardation. Mixtures of carbonic acid and acetylene have critical temperatures between those of the component pure gases. The diagram connecting temperature and volume shows that the plait-point curve is a line with small curvature; the border-curve is relatively narrow. An instance of a critical temperature above those of the components, for this mixture, has not yet been proved. Theory indicates that this phenomenon probably occurs for mixtures having a minimum vapour-pressure at low temperatures. Critical temperatures below those of components, seem to occur for mixtures having a maximum vapour-pressure, as for nitrous oxide and ethane. The law connecting the two phenomena is deduced from van der Waal's theory. As a further application of this theory, it is shown that in consequence of certain coincidences between the real border-curve and the hypothetical border-curve, the critical point of the maximum mixture may be determined in exactly the same way as for a single substance. A remark is added with regard to the condensation of such substances as exhibit changes of molecular systems. If an association takes place of molecules to more complicated systems, van der Waal's formula does not apply.

Dr. S. P. THOMPSON asked whether diagrams characteristic of cyanogen had been obtained. Its remarkable polymerism suggested an interesting case for critical phenomena.

Dr. KUENEN thought such a substance might be worth investigating.

A paper by Dr. BARTON, on "The Attenuation of Electric Waves in Wires," was taken as read.

Mr. G. F. C. SEARLE read a paper on "The Steady Motion of an Electrified Ellipsoid."

The first part of the investigation is printed in the *Phil. Trans. Roy. Soc.* It contains the principles required in the solution of problems with respect to moving electrical

charges. The second part, now presented to the Physical Society, deals with the motion of a charged ellipsoid; the treatment is entirely mathematical. When any system of electric charges moves with uniform velocity through the æther, the electro-magnetic field, referred to axes moving forward with the charges, can be completely defined by means of a quantity of which the electric force and the magnetic force are simple functions. Another vector concerned in the problem is the mechanical force experienced by a unit charge moving with the rest of the system. A distribution of electricity on the surface of a charged body, such as to give zero distribution at all points inside the surface, is an equilibrium distribution. Since the mechanical force vanishes inside the surface, it is shown that on the outside of the surface the mechanical force is perpendicular to the surface, and the above-mentioned function is constant over the surface, and the distribution on an ellipsoid is the same for motion as for rest. When a charged sphere is at rest it produces the same effect as a point-charge at its centre. If the sphere is in motion, it produces the same effect as an uniformly-charged line whose length bears to the diameter of the sphere the same ratio that the velocity of the sphere bears to the velocity of light. When the sphere moves with the velocity of light, the line becomes the diameter of the sphere; the same is true for an ellipsoid. At the velocity of light the charge on any surface is in equilibrium, whatever the distribution. The force between two charges moving with the speed of light is zero. The lines of electric force for a charged sphere in motion are not radial; they form a series of hyperbolas. The author proceeds to calculate the total energy possessed by an ellipsoid when in motion along its axis of figure. Expressions are given (1) for the energy of a Heaviside ellipsoid; (2) for a sphere; and (3) for a very slender ellipsoid. In all cases the energy becomes infinite when the charges move at the velocity of light. It would seem impossible to make a charged body move at a greater speed than that of light.

Prof. PERRY said the paper would help to solve many problems connected with the effect of the rotation of the earth upon electrical surface changes. An expression might be found for the mechanical and magnetic forces due to the motion of a charge at any point of the earth's surface. At the equator a point moves at different velocity at midday to its midnight velocity; it might now be possible to determine the magnetic and mechanical effects due to electric charges at equatorial points.

Mr. BLAKESLEY asked whether, in calculating the mutual action of two charged particles, proceeding at the velocity of light, it was assumed that the lines of motion were parallel.

Mr. SEARLE said he had always considered parallel lines of motion; he could not say whether the force would be zero in any other case. The results arrived at in the paper could be applied to problems connected with distributions of terrestrial charges.

The PRESIDENT proposed votes of thanks to the authors; the meeting then adjourned until November.

OBITUARY.

PROF. SCHÜTZENBERGER.

CHEMICAL Science, not merely in France but throughout the world, has sustained a heavy blow by the death of Prof. Schützenberger, at the age of 67. The deceased was a native of Strasburg, where he studied medicine, and became connected with the chemical laboratory of the Conservatoire des Arts et Metiers, of that city. He became successively Assistant-Director of the Sorbonne Laboratory and Head of the Chemical Department of the Collège de France, where he filled the chemical chair since 1876. Along with these posts, which he held with

success and distinction, he was elected Head of the Paris Municipal School of Chemistry and Physics. In 1884 he became a Fellow of the Academy of Medicine. Lastly, in 1888, on the death of Débray, he was elected a Fellow of the Academy of Sciences.

These honours, and the widely-felt appreciation of which they were the embodiment, were earned by a series of sterling researches in organic chemistry, especially in its application to colours and their uses in the tinctorial arts, in which department he was recognised as one of the best authorities. His works include researches on the physiology of digestion and fermentation, and, above all, a cyclopædia—as we may call it—of tinctorial chemistry, considered both theoretically and practically, and an investigation into the alkaloids.

The late professor was universally respected by all who had the pleasure of his acquaintance. Prof. Schützenberger died on Monday, June 28th.

NOTICES OF BOOKS.

Fresenius's Quantitative Analysis. Vol. ii. Translated by C. E. GROVES, F.R.S. Part IV.

THE *magnum opus* of the late "Grand Master" (Professor Fresenius) is gradually making its appearance in an English guise.

The present issue treats in succession of zinc-dust, of manganese compounds, nickel compounds, iron compounds, uranium, silver, mercury, and lead compounds.

The translator appears to be doing justice to the original. At the same time we cannot approve of the system of publishing the translation of a book in detached portions.

The Study of Technical Chemistry at the Universities and Technical High Schools of Germany. ("Das Studium der Technischen Chemie am den Universitäten und Technischer Hoch-Schulen, Deutschlands"). *Opinions on the German System of Training Chemists*. By Prof. Dr. FERD. FISCHER.

THIS pamphlet contains the opinions of eminent authorities as to the cause of the relative decline of certain English industries. As a whole, they agree that our failure—for as such we must accept it—is due to our imperfect system of higher education. Chemical technology is for the universities the connecting link between science and industry.

Among the heads of German universities there prevails a unanimous opinion that Germany can maintain its industrial work solely in virtue of its chemical eminence. Slight alterations in the curriculum are proposed in some quarters. But the general tendency is rather to intensify than to lighten the course of study.

In the Presidential Address of the Society of Chemical Industry (1895), it was admitted that business ability and capital cannot suffice to uphold the chemical industry of the country without a fundamental scientific technical training.

We have met with chemical manufacturers who know little of chemical science, and do not seek to know more. Some of these employ an analyst who has to limit himself to routine work. To enquire into the cause of any disappointment is a "waste of time."

Chilian Hygienic Review. ("Revista Chilena de Higiene," publicada el Instituto de Higiene de Santiago). Parts II. and III.

THE most important matter in these issues refers to the observance of quarantine, which, in the whilome Spanish republics, is still maintained. In Chili there are two

grades of quarantine. The quarantine of observation is imposed upon every traveller upon entering Chilian territories, which lasts forty-eight hours. During this period the traveller and his belongings are submitted to systematic disinfection according to the rules laid down.

At the moment when the quarantine expires the traveller will receive, from the head of the sanitary department, a certificate which will serve him as a passport. The head of the sanitary department will communicate with the governor of the department to which the traveller is proceeding.

Rigid Quarantine.—If, during the quarantine of observation, there occur suspicious symptoms, the quarantine is made strict. Rigid quarantine is then continued for eight days. If the patient dies, his body, after disinfection, is buried in ground the drainage of which does not enter the stream. If no such ground is obtained, the body will be incinerated. His possessions will be destroyed by fire, with the exception of articles which the head of the sanitary department may designate.

The disinfection of persons will consist in a general soap-bath or in general lotions, with a solution of sublimate at 1/5000.

Dead bodies will receive an injection of solution of sublimate at 1/500, in the stomach, the bowels, and the carotid and femoral arteries, and the body will further be wrapped in a shroud saturated with the same solution.

CORRESPONDENCE.

ESTIMATION OF CARBON IN FERRO-CHROME.

To the Editor of the Chemical News.

SIR,—Professor Arnold's letter is a surprising continuation of what I hoped might prove a helpful discussion. I ought first to disabuse your readers' mind of the personal illusion by which the Professor would create a false relation between us. He says that "until quite recently Mr. Leffler was a student of mine." In the ordinary sense this is not true. It is true that I was a student at the Technical School for three years, but it is nine years this summer since I left; that was some time before Prof. Arnold came to the school. This he can readily verify by appealing to his Senior Demonstrator. In the autumn of 1894 I desired to acquaint myself with the microscopic analysis of steel, and worked under Prof. Arnold two nights a week from September to Christmas, and ceased work at his suggestion on account of the inadequate accommodation. This fact is a very slender thing on which to found the above assertion, and I feel that it in no way obliges me to submit to him any opinion I may form on a subject like the one under discussion.

I pointed out in my last letter that I had never made the bald assertion that his method only gave half the carbon present. The furnace at our disposal certainly only gave about 50 per cent of the carbon, but I stated that on raising the temperature with the blowpipe the remaining 50 per cent was obtained. Plainly, then, our furnace was not hot enough, though it was as hot as combustion furnaces frequently are. The need of the blowpipe was evidence that the temperature was too low, and I have never attempted to deny it.

Prof. Arnold invited a reply to his first letter, by asking the type of furnace and the diameter of the gas-pipe. To this information I attached a few questions and other details, which the Professor has no inclination to follow, although I refer only to certain pages of his own book, "Steel Works Analysis."

Prof. Arnold refers to Mr. Saniter's letter as proof that I have put up straw men. I cannot imagine to what part he refers, but thank him for the reference nevertheless, because it draws my attention to the unprejudiced support Mr. Saniter gives to my assertion. Saniter's third para-

graph reads "I also found that with copper oxide only about half the carbon is obtained." Now I admit that a little higher temperature is needed for combustion with copper oxide than with lead chromate; but copper oxide will give perfectly accurate results, and failed to do so in Mr. Saniter's hands only because his furnace was not hot enough, which supports my assertion that our furnace is as hot as those generally used, but not enough to completely eliminate the carbon from a mixture of lead chromate and ferro-chrome.

The statement made was not inaccurate, nor was it hastily arrived at, as the method was given a long and fair trial, and the results obtained accurately and honestly recorded.—I am, &c.,

R. L. LEFFLER.

The Laboratory,
Messrs. Thos. Firth and Sons, Lim.,
Sheffield, June 21, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 22, May 31, 1897.

Liquefaction of Fluorine.—H. Moissan and J. Dewar.—This memoir has been inserted in full.

Part Played by Humic Matter in the Fertilisation of Soils.—Armand Gautier.—The algae as well as the microbia which solidify nitrogen find in the humus of the soil and generally in the organic matter of excrements a nutrient which allows of their rapid development. I do not assert that soils owe their fertility to the direct absorption by plants of organic matter, ternary or quaternary.

Purification of Cerium.—M. Wyruboff and A. Verneuil.—Already inserted.

Remarks on the above Communication by Wyruboff and Verneuil.—Already inserted.

Alloys of the Silver-copper Group.—F. Osmond.—The conception of Matthiessen, who saw on certain alloys solidified solutions of allotropic form, whilst in need of certain restrictions, seems to remain vital and fruitful. I do not see that it is opposed to the researches of H. le Chatelier, whose conclusions would receive from it a character of greater generality.

Phosphorescence of Strontium Sulphide.—Joaquín Rodríguez Mourello.—We see that an oxidising principle is requisite, as well as a peculiar structure, for strontium sulphide to be susceptible of phosphorescence without omitting the substances whose influence on the property in question is direct or positive.

Contribution to the Study of the Preparation of Common Ether.—L. Prunier.—In the ordinary preparation of ether it escapes, in virtue of its great volatility, in the midst of a heterogeneous medium, unstable, and in perpetual transformation.

Certain Compounds of Phenylhydrazine with Metallic Chlorides.—J. Ville and J. Moitessier.—Phenylhydrazine combines with different metallic chlorides, yielding compounds containing 1 mol. of chlorides with 2 mols. of phenylhydrazine.

Apparatus for the Industrial Analysis of Gases.—Leo Vignon.—This memoir requires the accompanying illustration.

Decomposition-products of Calcium Carbide, and on its Use as an Insecticide.—E. Chuard.—The author has obtained a phospho-carbide possessing powerful insecticide properties.

No. 23, June 8, 1897.

Action of Light on Mixtures of Chlorine and Hydrogen.—Armand Gautier and H. Hélier.—This paper will be inserted at some length.

Observations on the Limitation of Chemical Reactions, with reference to Armand Gautier's Communication.—This memoir will also be inserted *in extenso*.

Memoir by M. Berthelot accompanying the Presentation of his work on Thermo-chemistry.—A kind of preface to the author's recent works.

Examination of certain Spectra.—Lecoq de Boisbaudran.—A controversial paper, directed against Dr. Eder and Valenta. Except the small ray 563·8, seen with K_2SO_4 , but not with K_2CO_3 , all the rays of the author's diagram are seen with K_2SO_4 and K_2CO_3 , and have not been observed with the salts of Na.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Annual Consumption of Chemicals.—I should be extremely obliged if any reader would kindly let me know what is the average amount of sulphate of barium and sulphurite of sodium consumed annually in this country, and in what industries these products are chiefly used.—L. ALSATINI.

TO CORRESPONDENTS.

Eugene Achermann.—Please forward address; a letter awaits you.

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THOMAS W. SHORE, Warden.

PATENTS, DESIGNS, AND TRADE MARKS ACTS, 1883 TO 1888.

NOTICE IS HEREBY GIVEN, that HIPPOLYTE EUGENE SERULLAS, of 20, Rue Moliere, Paris, France, has applied for leave to amend the Specification of the Letters Patent No. 11166 of 1892 for "Improvements in or connected with an Apparatus for the Obtaining or Extraction of Gutta-percha or the like."

Particulars of the proposed amendments were set forth in the Illustrated Official Journal (Patents), issued on the 16th June, 1897.

Any person, or persons, may give notice of opposition to the amendment (on Form G) at the Patent Office, 25, Southampton Buildings, London, W.C., within one calendar month from the date of the said Journal.

(Signed) C. N. DALTON,
Comptroller General.

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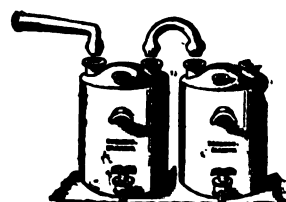
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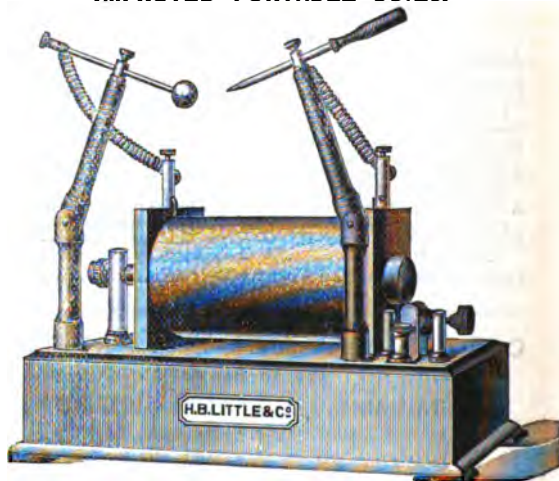
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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1963.

DIAMONDS.*

By WILLIAM CROOKES, F.R.S., M.R.I.

(Continued from p. 4).

Conversion of Diamond into Graphite.

I WILL now draw your attention to a strange property of the diamond, which at first sight might seem to argue against the great permanence and unalterability of this stone. It has been ascertained that the cause of phosphorescence is in some way connected with the hammering of the gaseous molecules, violently driven from the negative pole on to the surface of the body under examination, and so great is the energy of the bombardment, that impinging on a piece of platinum, or even iridium, the metal will actually melt. When the diamond is thus bombarded in a radiant matter tube the result is startling. It not only phosphoresces, but assumes a brown colour, and when the action is long continued becomes almost black.

I will project a diamond on the screen and bombard it with radiant matter before your eyes. I do not like to anticipate a failure, but here I am entirely at the mercy of my diamond. I cannot rehearse this experiment beforehand, and it may happen that the diamond I have selected will not blacken in reasonable time. Some visibly darken in a few minutes, while others, more leisurely in their ways, require an hour.

This blackening is only superficial, but no ordinary means of cleaning will remove the discolouration. Ordinary oxidising reagents have little or no effect in restoring the colour. The black stain on the diamond is due to a form of graphite which is very resistant to oxidation. It is not necessary to expose the diamond in a vacuum to electrical excitement in order to produce this change.

I have already signified that there are various degrees of refractoriness to chemical reagents among the different forms of graphite. Some dissolve in strong nitric acid; other forms of graphite require a mixture of highly concentrated nitric acid and potassium chlorate to attack them, and even with this intensely powerful agent some graphites resist longer than others. M. Moissan has shown that the power of resistance to nitric acid and potassium chlorate is in proportion to the temperature at which the graphite was formed, and with tolerable certainty we can estimate this temperature by the resistance of the specimen of graphite to this reagent.

The superficial dark coating on a diamond after exposure to molecular bombardment I have proved to be graphite,† and M. Moissan‡ has shown that this graphite, on account of its great resistance to oxidising reagents, cannot have been formed at a lower temperature than 3600° C.

It is therefore manifest that the bombarding molecules, carrying with them an electric charge, and striking the diamond with enormous velocity, raise the superficial layer to the temperature of the electric arc, and turn it into graphite, whilst the mass of diamond and its conductivity to heat are sufficient to keep down the general temperature to such a point that the tube appears scarcely more than warm to the touch.

A similar action occurs with silver, the superficial layers of which can be raised to a red heat without the whole mass becoming more than warm.§

This conversion of diamond into graphite is I believe a pure effect of heat. In 1880* Professor Dewar in this theatre placed a crystal of diamond in a carbon tube through which a current of hydrogen was maintained. The tube was heated from the outside by an electric arc, and in a few minutes the diamond was converted into graphite. I will now show you that a clear crystal of diamond, heated in the electric arc (temperature 3600° C.) is converted into graphite, and this graphite is most refractory.

The diamond is remarkable in another respect. It is extremely transparent to the Röntgen rays, whereas highly refracting glass, used in imitation diamonds, is almost perfectly opaque to the rays. I exposed over a photographic plate to the X rays for a few seconds the large Delhi diamond, of a fine pink colour, weighing 3½ carats, a black diamond weighing 23 carats, together with an imitation in glass of the pink diamond lent me by Mr. Streeter; also a flat triangular crystal of diamond of pure water, and a piece of glass of the same shape and size. On development, the impression where the diamond obscured the rays was found to be strong, showing that most rays passed through, while the glass was practically opaque. By this means imitation diamonds and some other false gems can readily be detected and distinguished from the true gems. It would take a good observer to distinguish my pure triangular diamond from the adjacent glass imitation.

Genesis of the Diamond.

Speculations as to the probable origin of the diamond have been greatly forwarded by patient research, and particularly by improved means of obtaining high temperatures. Thanks to the success of Professor Moissan, whose name will always be associated with the artificial production of diamonds, we are able to-day to manufacture diamonds in our laboratories—minutely microscopic, it is true—all the same veritable diamonds, with crystalline form and appearance, colour, hardness, and action on light the same as the natural gem.

Until recent years carbon was considered absolutely non-volatile and infusible; but the enormous temperatures at the disposal of experimentalists—by the introduction of electricity—show that, instead of breaking rules, carbon obeys the same laws that govern other bodies. It volatilises at the ordinary pressure at a temperature of about 3600° C., and passes from the solid to the gaseous state without liquefying. It has been found that other bodies which volatilise without liquefying at the ordinary pressure will easily liquefy if pressure is added to temperature. Thus, arsenic liquefies under the action of heat if the pressure is increased; it naturally follows that if along with the requisite temperature sufficient pressure is applied, liquefaction of carbon will be likely to take place, when on cooling it will crystallise. But carbon at high temperatures is a most energetic chemical agent, and if it can get hold of oxygen from the atmosphere or any compound containing it, it will oxidise and fly off in the form of carbonic acid. Heat and pressure therefore are of no avail unless the carbon can be kept inert.

It has long been known that iron when melted dissolves carbon, and on cooling liberates it in the form of graphite. Moissan discovered that several other metals have similar properties, especially silver; but iron is the best solvent for carbon. The quantity of carbon entering into solution increases with the temperature, and on cooling in ordinary circumstances it is largely deposited as crystalline graphite.

Professor Dewar has made a calculation as to the Critical Pressure of Carbon—that is, the lowest pressure at which carbon can be got to assume the liquid state at its critical temperature, that is the highest temperature at which liquefaction is possible. He starts from the

* A Lecture delivered at the Royal Institution, Friday, June 11th, 1897.

† *Chemical News*, vol. lxxiv., p. 39, July, 1896.

‡ *Comptes Rendus*, cxxiv., p. 653.

§ *Proc. R. S.*, vol. 1., p. 99., June 1891.

* *Proceedings of the Royal Institution*, Friday Evening Meeting, Jan. 16, 1880.

vaporising or boiling point of carbon, which, from the experiments of Violle and others on the electric arc, is about 3600°C. , or $3874^{\circ}\text{Absolute}$. The critical point of a substance on the average is 1.5 times its absolute boiling point. Therefore the critical point of carbon is 5811°Ab. , or, say, 5800°Ab. But the absolute critical temperature divided by the critical pressure is for elements never less than 2.5. Then—

$$\frac{5800^{\circ}\text{A.}}{\text{PCr}} = 2.5, \text{ or } \text{PCr} = \frac{5800^{\circ}\text{A.}}{2.5}, \text{ or } 2320 \text{ atmospheres.}$$

The result is that the critical pressure of carbon is about 2300 atmospheres, or say 15 tons on the square inch. The highest critical pressure recorded is that of water, amounting to 195 atmospheres, and the lowest that of hydrogen, about 20 atmospheres. In other words, the critical pressure of water is ten times that of hydrogen, and the critical pressure of carbon is ten times that of water.

Now 15 tons on the square inch is not a difficult pressure to obtain in a closed vessel. In their researches on the gases from fired gunpowder and cordite, Sir Frederick Abel and Sir Andrew Noble obtained in closed steel cylinders pressures as great as 95 tons to the square inch, and temperatures as high as 4000°C. Here, then, if the observations are correct, we have sufficient temperature and enough pressure to liquefy carbon; and if the temperature could only be allowed to act for a sufficient time on the carbon there is little doubt that the artificial formation of diamonds would soon pass from the microscopic stage to a scale more likely to satisfy the requirements of science, industry, and personal decoration.

Artificial Manufacture of the Diamond.

I now proceed to manufacture a diamond before your eyes—don't think I yet have a talisman that will make me rich beyond the dreams of avarice! Hitherto the results have been very microscopic, and are chiefly of scientific interest in showing us Nature's workshop, and how we may ultimately hope to vie with her in the manufacture of diamonds. Unfortunately the operations of separating the diamond from the iron and other bodies with which it is associated are somewhat prolonged—nearly a fortnight being required to detach it from the iron, graphite, and other matters in which it is embedded. I can, however, show the different stages of the operations, and project on the screen diamonds made in this manner.

In Paris recently I saw the operation carried out by M. Moissan, the discoverer of this method of making carbon separate out in the transparent crystalline form, and I can show you the operations straight as it were from the inventor's laboratory. I am also indebted to the Directors of the Notting Hill Electric Lighting Co. and to the General Manager, Mr. Schultz, for enabling me to perform several operations at their central station, where currents of 500 amperes and 100 volts were placed at my disposal.

The first necessity is to select pure iron—free from sulphur, silicon, phosphorus, &c.,—and to pack it in a carbon crucible with pure charcoal from sugar. Half a pound of this iron is then put into the body of the electric furnace, and a powerful arc formed close above it between carbon poles, utilising a current of 700 amperes at 40 volts pressure. The iron rapidly melts and saturates itself with carbon. After a few minutes' heating to a temperature above 4000°C. —a temperature at which the lime of the furnace melts like wax and volatiles in clouds—the current is stopped, and the dazzling fiery crucible is plunged beneath the surface of cold water, where it is held till it sinks below a red heat. As is well known, iron increases in volume at the moment of passing from the liquid to the solid state. The sudden cooling solidifies the outer layer of iron and holds the inner molten mass in a tight grip. The expan-

sion of the inner liquid on solidifying produces an enormous pressure and under the stress of this pressure the dissolved carbon separates out in a transparent, dense crystalline form—in fact, as diamond.

Now commences the tedious part of the process. The metallic ingot is attacked with hot nitro-hydrochloric acid until no more iron is dissolved. The bulky residue consists chiefly of graphite, together with translucent flakes of a chestnut-coloured carbon, black opaque carbon of a density of from 3.0 to 3.5, and hard as diamonds—black diamonds or carbonado, in fact—and a small portion of transparent colourless diamonds showing crystalline structures. Besides these, there may be carbide of silicon and corundum, arising from impurities in the materials employed.

The residue is first heated for some hours with strong sulphuric acid at the boiling point, with the cautious addition of powdered nitre. It is then well washed and allowed for two days to soak in strong hydrofluoric acid in the cold, then in boiling acid. After this treatment the soft graphite will disappear, and most if not all of the silicon compounds will be destroyed. Hot sulphuric acid is again applied to destroy the fluorides, and the residue, well washed, is repeatedly attacked with a mixture of the strongest nitric acid and powdered potassium chlorate, kept warm, but to avoid explosions not above 60°C. This ceremony must be repeated six or eight times, when all the hard graphite will gradually be dissolved, and little else left but graphitic oxide, diamond, and the harder carbonado and boart. The residue is fused for an hour in fluorhydrate of fluoride of potassium, then boiled out in water, and again heated in sulphuric acid. The well washed grains which resist this energetic treatment are dried, carefully deposited on a slide, and examined under the microscope. Along with numerous pieces of black diamond are seen transparent colourless pieces, some amorphous, others with a crystalline appearance, as I have attempted to reproduce in diagrams. Although many fragments of crystals occur, it is remarkable that I have never seen a complete crystal. All appear broken up, as if on being liberated from the intense pressure under which they were formed they burst asunder. I have direct evidence of this phenomenon. A very fine piece of artificial diamond, carefully mounted by me on a microscopic slide, exploded during the night and covered my slide with fragments. This bursting paroxysm is not unknown at the Kimberley mines.

On the screen I will project fragments of artificial diamond, some lent me by Professor Roberts-Austen, others of my own make; while on the wall you will see drawings of diamonds copied from M. Moissan's book on the Electric Furnace. Unfortunately these specimens are all microscopic. The largest artificial diamond, so far, is less than one millimetre across.

Laboratory diamonds burn in the air before the blow-pipe to carbonic acid; and in lustre, crystalline form, optical properties, density, and hardness they are identical with the natural stone.

Many circumstances point to the conclusion that the diamond of the chemist and the diamond of the mine are strangely akin as to origin. It is conclusively proved that the diamond has not been formed *in situ* in the blue ground. The diamond genesis must have taken place at great depths under enormous pressure. The explosion of large diamonds on coming to the surface shows extreme tension. More diamonds are found in fragments and splinters than in perfect crystals; and it is noteworthy that although many of these splinters and fragments are derived from the breaking up of a large crystal yet in no instance have pieces been found which could be fitted together. Does not this fact point to the conclusion that the blue ground is not their true matrix? Nature does not make fragments of crystals. As the edges of the crystals are still sharp and unabraded, the locus of formation cannot have been very distant from the present sites. There were probably many sites of

crystallisation differing in place and time, or we should not see such distinctive characters in the gems from different mines, nor indeed in the diamonds from different parts of the same mine.

(To be continued).

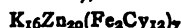
ON THE VOLUMETRIC DETERMINATION OF ZINC BY POTASSIUM FERROCYANIDE.

By L. L. DE KONINCK and EUG. PROST.

(Continued from p. 6).

In fact, by running, to the exact precipitation point, a solution of ferrocyanide into a measured volume of zinc solution acidulated with sulphuric acid, he asserts that there is required, for 25 c.c. of zinc solution, not 12.5 c.c. of ferrocyanide, but 17.75 c.c. to 19.5 c.c., according to the acidity and concentration of the mixture.

The volume of ferrocyanide used diminishes as the acidity increases, and also with the dilution of the zinc solution. According to Zulkowski it would be quite indifferent whether the zinc solution is run into the potassium ferrocyanide or *vice versa*. By boiling the liquid, constant results can be obtained independent of the acidity, and the precipitate—which is then pulverulent instead of gelatinous—will have a constant composition.* Zulkowski recognised the end of his tests by the formation of Prussian blue, by placing side by side on a piece of filter-paper a drop of the mixture and a drop of ferric chloride. By taking note of the excess of ferrocyanide necessary to show with the ferric chloride (0.4 c.c. for each 85 c.c. of the mixture), he concludes that—



is the correct formula for the precipitate obtained by heating, and $K_{16}Zn_{12}(Fe_2Cy_{12})_5$ for that obtained in the cold, but he is not very certain of the latter.

These complicated formulæ seem at first sight improbable,—we shall show, in fact, that they ought to be rejected; the estimations or calculations on which they are based are full of errors.†

F. Regelsberger (*Zeitsch. f. Angew. Ch.*, vol. iv., p. 475, 1891), when analysing alloys of aluminium, quotes the estimation by ferrocyanide as very rapid and quite sufficiently exact.

We will here refer again to the work of Bein and Bragard. The former, who evidently had not the experience necessary to judge the value of volumetric methods to a nicety, does not speak well either of Schaffner's method or of the Galletti-Fahlberg, but prefers the gravimetric method.

Bragard gives the ferrocyanide precipitate the composition answering to the formula $K_2Zn_3Fe_2Cy_{12}$ when the reaction is at an end, in which we think he is right; but when the zinc is in excess the composition would be remedied by the formula $K_4Zn_{10}(Fe_2Cy_{12})_3$; here we think he is mistaken. According to him the precipitate would react with the nitrate of uranium used as indicator. He also interposes a sheet of filter-paper between the rod carrying the trial drop and the paper impregnated with uranium salt, and does not consider the reaction at an end while an appreciable colouration is produced on this second paper. In our opinion he thus diminishes the sensitiveness of the indicator; also, according to him, the process is not exact to less than $\frac{1}{4}$ per cent. The acidity of the liquid, he says, influences the result, but not the

dilution, of the presence of ammonia,—he evidently means salts of ammonia.

To complete our references to work which has already been published, we must again mention the Report made to the Society of Colorado. The Committee charged with examining into the methods employed in this important mining district sent to several chemists five samples of minerals, carefully selected, and analysed with every possible precaution, begging them at the same time to send, with their results, the details of the process used. All of them titrated the acid solution with ferrocyanide, using a salt of uranium as indicator. The results were as follows:—

No.	A.	B.	C.	D.	E.	F.*	G.
1.	15.31	15.39	15.66	15.08	14.62	—	14.38 14.22
2.	24.34	24.53	24.23	23.80	22.00	23.62	22.95 23.11
3.	10.76	10.83	11.88	10.69	10.50	11.07	8.58 9.20
4.	6.42	6.58	8.73	6.85	6.30	6.89	5.24 5.64
5.	16.14	16.46	15.86	15.90	15.37	16.08	13.40 12.84

* Average of four concordant assays. No. 1 was not analysed.

The actual results obtained gravimetrically were:—

14.64 24.11 10.71 6.31 16.09.

The samples 1 and 2, but especially No. 1, contained cadmium in appreciable quantity, which will account for the generally high results, this metal not having been eliminated. It is worthy of remark that they do not as a rule use hydrosulphuric acid; when the mineral contains copper they precipitate it by means of granulated lead, which is well shaken up in the acid solution.

We may conclude from these results that the process will furnish exact results, so long as it is carried out under specified conditions and with the necessary care.

III. Reaction of Ferrocyanide of Potassium with Zinc Salts in Acid Solution.

As we have seen, there is very little accord as to the composition of the precipitate, which is formed in an acid zinc solution, on the addition of ferrocyanide.

All admit that if ferrocyanide be added in sufficient quantity for it to be in slight excess, and to be detected in the mixture by a uranium salt, or by other means, the quantity employed is notably more than that which is necessary theoretically to produce a simple zinc ferrocyanide, $Zn_4Fe_2Cy_{12}$. Nevertheless some chemists give this formula to the precipitate and explain away the difference, although it is considerable, by impurities in the substance used.

The authors of this paper, who have studied ferrocyanides, have long been aware that the zinc precipitate obtained by an excess of ferrocyanide is a double salt, $K_2Zn_3Fe_2Cy_{12}$ (Mosander, *Handw. d. Reinen u. Angew. Chem.*, p. 867, 1848; F. Reindel, *Polyt. Journ. de Dingler*, vol. cxc., p. 395, 1869; G. Wyruboff, *Ann. de Chim. et de Phys.*, Series 5, vol. viii., p. 444, 1876); that has not prevented many other analysts attributing to it much more complicated formulæ. This question of the composition of the precipitate,—that is to say, the relation between the zinc and the ferrocyanide,—being of the first importance to us, it was imperative to decide the point definitely, once and for all; in this we have been successful. Our work has settled the point in dispute.

In fact, from the first titrations done by us, to get some idea of the true value of the ferrocyanide process, as described by most writers (direct titration in acid solution), we found, by running a decinormal solution* of ferrocyanide into 25 c.c. of a similarly decinormal solution of zinc chloride,† made slightly acid and diluted with water

* We shall see later that this transformation is of great importance.

† The author must have made a mistake in his calculations; the reaction, in presence of ferrocyanide of potassium in excess, is very sharp, and the relation between the potassium and the zinc is 2 to 3, or 8 to 12, and not 16 to 12.

* The molecular weight of $K_2Fe_2Cy_{12} \cdot 6H_2O$, being 843.52, the normal weight is represented by $\frac{1}{10}$ of this figure, viz., 84.352. Our decinormal solution was prepared by dissolving 52.72 grms. of pure ferrocyanide in 1 litre of water.

† Obtained by dissolving 16.2775 grms. of pure zinc in the smallest possible quantity of hydrochloric acid, and then diluting to 1 litre.

to 150 c.c., that, working with from 26 to 28 c.c. of ferrocyanide, we obtained, by testing immediately with uranium salts,* an appreciable brown colouration, which does not get stronger by the addition of ferrocyanide, which is further less sensitive as we increase the time from the addition of the titrate and the touch test, and eventually disappears if this time be sufficiently prolonged. But with from 33·3 to 33·5 c.c. the test becomes more and more distinct, and that no matter what time elapses before the addition of the ferrocyanide. These facts can be explained in several different manners.

The most plausible which first occurred to us is the following:—When the ferrocyanide is added to the zinc solution, a ferrocyanide of zinc, $Zn_4Fe_2Cy_{12}$, is first formed, so long as the zinc salt is in excess. The formula, $4ZnCl_2 + K_2Fe_2Cy_{12} = 8KCl + Zn_4Fe_2Cy_{12}$, would therefore show what takes place during the first part of the reaction,—that is to say, in our experiment, up to the addition of 25 c.c. of ferrocyanide.

By continuing to add this reagent, the alkaline ferrocyanide would then reach more or less slowly with the zinc precipitate, forming the double ferrocyanide, $K_2Zn_3Fe_2Cy_{12}$, according to the formula—



This reaction is not immediate; the mixture will react with the nitrate of uranium, while neither the zinc ferrocyanide nor the double ferrocyanide do.

Certain tests, to which we shall refer later on, incline us to the belief that the double cyanide is really formed in the first instance; that it, being gelatinous at first, can in this condition react with the uranium salt so long as it is not in presence of a large excess of zinc; but that it undergoes a molecular transformation which shows itself by passing from the translucent gelatinous state to a more coherent granular state, in which it will not act with the indicator. Perhaps both theories are correct,—that is to say, the phenomena may be produced simultaneously. It is in any case proved that the indicator does not show, in a definite manner, that when the volume of ferrocyanide used, corresponds sensibly to the formation of the double salt, and that this latter, the same as with the simple zinc ferrocyanide, if there be one formed, will not react with the nitrate of uranium after a sufficient digestion. It is equally shown that an excess of ferrocyanide of potassium does not combine with the double salt, $K_2Zn_3Fe_2Cy_{12}$, at least not under the conditions of our experiments, which were carried out with a deminormal solution of zinc chloride and a solution, $\frac{1}{2}$ normal for zinc,† of ferrocyanide.

We here give the result. In each case we used 25 c.c. of $ZnCl_2$, diluted with 100 c.c. of water and acidulated with hydrochloric.

A. Add 29 c.c. of ferrocyanide. The zinc is now in excess, even allowing the formation of $Zn_4Fe_2Cy_{12}$, which would require 37·5 c.c. for complete precipitation. The precipitate, gelatinous at first, becomes gradually white and flocculent; it collects and deposits in less than fifteen minutes.

B. Add 38 c.c. of ferrocyanide, that is, 0·5 c.c. more than the quantity necessary to precipitate the zinc entirely, as $Zn_4Fe_2Cy_{12}$. The precipitate behaves in exactly the same way as in the preceding experiment. In the solution, cleared by sedimentation, a fresh addition of ferrocyanide produces an abundant precipitate: this proves the presence of zinc in solution, and it therefore follows that the first precipitate is not entirely composed of $Zn_4Fe_2Cy_{12}$ —if, indeed, it contains this compound. As the first precipitate, A, behaves exactly like the second one, we can only conclude that it also, in spite of the notable excess of chloride of zinc, is not $Zn_4Fe_2Cy_{12}$.

C. Add quickly 50 c.c. of ferrocyanide, that is, the

theoretical amount necessary to precipitate all the zinc as $K_2Zn_3Fe_2Cy_{12}$.

If tested immediately, the mixture gives a strong brown colouration with nitrate of uranium. If filtered at once, the mixture yields at first an absolutely limpid filtrate, which gives no colouration with uranium; it is therefore free from potassic ferrocyanide. After a few moments the filtrate becomes very cloudy, and finally becomes clear again. Neither the cloudy filtrate nor the final clear filtrate gives any colour with the indicator. A small quantity of the unfiltered mixture, put on one side, remains milky, and reacts very feebly with nitrate of uranium.

D. Add quickly, while agitating, 50 c.c. of ferrocyanide. An immediate test of the mixture with nitrate of uranium gives an intense brownish red colouration; after half a minute, a colour of "café-au-lait;" after a minute, a clear coffee-colour; after another half minute, a very feeble reaction; after two minutes, nothing at all. Then add 5 c.c. of ferrocyanide; the mixture will again show an intense brown colour when tested with uranium; the same result will be obtained after an hour, or even two hours.

(To be continued).

BASIC SALTS OF CADMIUM.

By M. TASSILLY.

THE action of metallic oxides, on the corresponding haloid salts, has enabled me to prepare two new compounds of cadmium, an oxybromide and an oxyiodide.

These bodies were obtained by heating a concentrated solution of bromide or iodide in the presence of oxide of cadmium, up to 200°, in a sealed tube. The quantities obtained were very small. These new bodies are distinctly crystalline and act on polarised light.

Analysis has given, for the oxyiodide, the formula CdI_2, CdO_3, H_2O .

	Found.	Calculated.
Iodine	$\begin{cases} 46\cdot45 \\ 46\cdot62 \end{cases}$	46·35
Cadmium	41·07	40·87

This compound is only slightly attacked by water. At 120° it does not vary in weight, either in nitrogen or in dry air freed from carbonic acid.

With parallel rays of light the crystals show extinction parallel to the longitudinal axis. In converging light we observe a lemniscate. The crystal is doubly refracting at very wide axes.

The oxybromide is in very small crystals, answering to the formula $CdBr_2, CdO_3, H_2O$; it also acts on polarised light.

	Found.	Calculated.
Bromine	36·44	35·24
Cadmium	48·90	49·33

I would recall the fact that M. Schulten (*Comptes Rendus*, vol. cvi., p. 1674) obtained both an oxychloride and an oxybromide, by the action of marble on chloride, or bromide of cadmium, at about 200°, in a sealed tube, both bodies being crystalline and answering respectively to the formulæ $CdCl_2, CdO, H_2O$ and $CdBr_2, CdO, H_2O$. Their stability when heated induced him to attribute to these bodies the constitutions—



M. Habermann (*Monatshette*, vol. v., p. 432, and *Bull. Soc. Chim.*, Ser. 2, vol. xlv., p. 122) obtained an amorphous oxychloride of the same formula by adding ammonia to a cold solution of chloride of cadmium as long as a precipitate was formed. It was then boiled, let stand, filtered, and the precipitate dried over quicklime under a bell-glass.

* One per cent solution of nitrate of uranium.

† We mean to infer, by the expression *normal for zinc*, a solution which will precipitate, volume for volume, a normal solution of zinc in the form $K_2Zn_3Fe_2Cy_{12}$.

By acting in the same manner on the bromide and the iodide, I obtained two compounds corresponding to definite formulæ; but the preparation of these bodies necessitates certain precautions. In fact, if we pour strong ammonia into solutions of chloride and bromide at 1/5th and of iodide at 1/7th strength, we obtain at the same time a basic salt and an ammoniacal salt which are very difficult to separate.

Working with more dilute solutions (1/10th) and adding ammonia equally diluted (ammonia at 22°, to which is added its own volume of water), we obtain first a precipitate of the basic salt, and some hours after decantation there forms in the mother-liquor crystals of the ammoniacal salt. One can even prevent altogether the formation of ammoniacal salts, by carefully limiting the proportion of ammonia.

The ammoniacal salts obtained under these conditions agree with the formulæ $\text{CdCl}_2 \cdot 2\text{NH}_3$; $\text{CdBr}_2 \cdot 2\text{NH}_3$; $\text{CdI}_2 \cdot 2\text{NH}_3$.

These bodies are identical with those obtained by dissolving the corresponding salts of cadmium in ammonia.

The oxybromide, $\text{CdBr}_2 \cdot \text{CdO} \cdot \text{H}_2\text{O}$, and the oxyiodide, $\text{CdI}_2 \cdot \text{CdO} \cdot \text{H}_2\text{O}$, gave on analysis the following results:—

<i>Oxybromide.</i>		
	Found.	Calculated.
Bromine..	37'17	38'29
Cadmium .	54'23	53'58
<i>Oxyiodide.</i>		
Iodine ..	49'77	49'63
Cadmium .	43'54	43'75

The basic salts of cadmium obtained by precipitation are decomposed by water.

I would here further remark that, in the action of ammonia on the salts of cadmium in solution, the proportions of basic salts obtained decrease from the oxychloride to the oxyiodide, while, on the contrary, the proportions of ammoniacal salts increase from the chloride to the iodide.

The chloride would thus appear to give a basic salt more easily than an ammoniacal one; the inverse should be the case for the iodide.

The thermo-chemical study of these various bodies, which I am now occupied with, will without doubt give the reason of these facts.

In finishing, it may be remarked that the basic salts of cadmium are always formed in equal molecules, contrary to what happens in the case of the salts of zinc, which are liable to fix a variable number of molecules of oxygen, giving rise to a number of salts not corresponding to a general type, as occurs with certain other metals.—*Bull. Soc. Chim. de Paris*, Series 3, vol. xvii.—xviii., No. 12.

HYPOIODOUS ACID AND HYPOIODITES.*

By R. L. TAYLOR, F.C.S.

Hypoiodites.

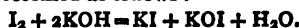
It appears to have been always considered very doubtful whether hypoiodous acid has ever been prepared at all, and many chemists are hardly willing to recognise hypoiodites as very definite compounds. The information one can obtain about these bodies is very vague and indefinite, and in some respects contradictory.

My investigation was originally undertaken with the object of isolating hypoiodous acid, but the following experiment led me to include hypoiodites as well. I had found that a solution of iodine in water acted in many respects very much better than any other solution, or

than the solid substance, and trying the effect of adding a little alkali to some of this aqueous solution, I was astonished at the particularly definite character of the solution obtained, and especially at its bleaching action, and felt sure that this remarkable solution could not be generally known, or else hypoiodites would certainly have met with better recognition than they have hitherto received.

So far as I am aware, the most important papers on hypoiodites have been those by Schönbein (*Journal für Praktische Chemie*, 1861, p. 387), and by G. Lunge and R. Schoch (*Berichte*, xv., p. 1883) on Calcium Hypoiodite. The more important of these is that by Schönbein, and in the first part of this paper I shall describe some of Schönbein's experiments, with others which I have performed and which confirm and extend his results. I shall refer to the work of Lunge and Schoch afterwards. I find that Schönbein, in his experiments, used the very solution which I have already mentioned as giving such remarkable results, that is, iodine dissolved in water. Unfortunately, however, Schönbein's paper has been badly summarised in all the standard dictionaries and works on chemistry, and this important point is not usually mentioned. Schönbein's paper is one of a series. He had been trying experiments on the action of chlorine water and bromine water upon dilute ammonia, and then naturally passed on to iodine, using that substance also in solution in water. Such a solution is very dilute, being at the most only about one part in 5000; but this solution, in many respects, gives more definite results than any other.

Schönbein first described the action of ammonia upon iodine water, whereby the liquid was decolourised, and a solution obtained which bleached indigo, just as the liquids produced by the action of ammonia upon chlorine water and bromine water did. He found, further, that the solution gave a deep blue colouration with a mixture of starch-paste and potassium iodide, and even with starch-paste alone. Left to itself the liquid lost these peculiarities, more quickly at high than at low temperatures, and almost instantaneously when boiled. He then found that similar results were obtained with potash solution, and that both solutions were decomposed by hydrogen peroxide, with manifest liberation of oxygen. He also pointed out that the solutions smelt of saffron. He not unnaturally concluded from these results that the liquids contained hypoiodites, and that the action of iodine upon the alkalies was similar to the action of chlorine and bromine, and might be represented as follows:—



He also concluded that, as the liquids lost their bleaching power, they gradually changed into iodide and iodate, according to the following equation:—



One thing which seemed to puzzle him very much was that the liquids gave a blue colour with starch alone, even when he added potash in the proportion of two equivalents to one of iodine. He thus made the liquid strongly alkaline, and capable, as he said, of taking up more iodine; and he argued, therefore, that there could not be any free iodine present in the excess of potash, and that hence the blue colour could not be due to iodine. In addition he pointed out that the liquid was almost, if not quite, colourless. He found, however, that the addition of potassium iodide turned the liquid brown again, manifestly owing to the liberation of iodine. I shall show further on that these results are easily explained.

I may mention that in all my experiments the iodine used was carefully purified by Stas's method, and that the indigo was a solution of indigo carmine in water.

As has been already mentioned, the liquids produced by the action of alkalies upon aqueous iodine have a most energetic bleaching action upon indigo; they also bleach cochineal and logwood, but not litmus. In bleaching

* From *Memoirs and Proceedings of the Manchester Literary and Philosophical Society*, vol. xli., Part III.

indigo they are much more active than either a solution of chlorine or of bleaching powder of anything like the same strength; in fact, compared with Schönbein's solutions, chlorine and hypochlorites may be described as very sluggish.

Borrowing, with some modifications, a method described by Lunge and Schoch in their paper, I attempted, by means of a standard solution of indigo carmine, to ascertain the strength of the bleaching liquids, in order to find, if possible,—assuming that the reaction goes as Schönbein suggested, and as the corresponding reaction with chlorine and bromine are well known to go,—the amount of iodine converted into what one may call "bleaching iodine." After many attempts I found that the best results were obtained by standardising the solution of indigo carmine against a dilute solution of chlorine, which had been titrated against a standard iodine solution by means of potassium iodide and sodium thiosulphate in the usual way. The aqueous iodine solution was also standardised against the same standard solution of iodine. The amount of iodine present in the aqueous solution was usually from 0.17 to 0.22 grm. per litre. One of the difficulties experienced in standardising the solutions was due to the end-reaction with chlorine water and the indigo solution being exceedingly slow. No such difficulty, however, was anticipated with the iodine bleaching solutions, the end-reaction with these being apparently sharp and distinct.

The method employed was to take a measured volume (usually 20 c.c.) of the aqueous iodine solution, to add one or two drops of potash or soda, and then immediately run in the standard indigo carmine until there was a distinct green colour. (The indigo solution is bleached to a slightly yellow liquid, and this of course becomes green as soon as an excess of indigo is added). For a long time the results were unsatisfactory. The bleaching power of the solutions seemed to vary in an extraordinary manner. Frequently the results obtained gave 90 and 95 per cent of the iodine converted into "bleaching iodine," and then, in another experiment, with the same solution of iodine, the bleaching action, without any apparent reason, ran up to 30 or even 40 per cent *above the theoretical amount*,—that is, above the amount which it ought to be if the whole of the iodine used had been converted into iodide and hypoiodite according to the equation—

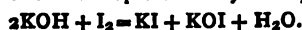


Similar anomalous results were obtained when solutions of bleaching powder or of sodium hypochlorite were used instead of chlorine water. These extraordinary results were ultimately found to be due to a very strange action on the part of the indigo, an action of which I can at present offer no explanation. The *excess* of bleaching action upon the indigo is not permanent; on standing for a minute or two the blue colour returns. This of course is not the case with what I may call the genuine bleaching action. If 1 c.c. of indigo solution in excess of what is permanently bleached be added, although there appears to be no indication of when the end-point is being passed, on standing for a minute or two a blue colour appears. I further found that this curious temporary bleaching action only occurs when a large excess of alkali (in comparison with the amount of iodine present) has been used.

Now, of course, it was possible to determine the amount of permanent bleaching action. The following example is one out of a great many experiments which I made:—

Twenty c.c. of the aqueous iodine solution (=0.0035 iodine), after the addition of alkali, bleached 15 c.c. of standard indigo solution, 1 c.c. of which (titrated with standard solution of chlorine) corresponded to 0.000228 of iodine, so that the amount of iodine indicated by the bleaching action was $0.000228 \times 15 = 0.00342$, which was practically the whole of the iodine. The solutions used are extremely dilute, but there is really no difficulty in

making estimations which will be accurate to within 2 or 3 per cent. The general result of these experiments is that 95 per cent of the iodine in Schönbein's solutions undergoes the reaction represented by the equation—



These results are amply confirmed by an altogether different method,—one which was used by A. Schwicker (*Zeit. Physikal. Chem.*, xvi., 303-314) in an investigation which he has recently made on the reaction velocity of potassium hypoiodite. He takes advantage of the fact that potassium bicarbonate will decompose a mixture of hypoiodite and iodide, with liberation of iodine. He also uses a little soda-water, the carbonic acid in which is intended to convert any liberated potash into the bicarbonate. The bicarbonate apparently decomposes the mixture of hypoiodite and iodide, with formation of normal carbonate and liberation of iodine, according to the following equation:—



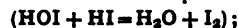
With my dilute solutions, I find that it answers just as well to run into the liquid, which is always sufficiently alkaline, a small quantity of soda-water. This immediately liberates the iodine, which can now be estimated by means of a centi-normal solution of sodium arsenite. Carbonic acid does not decompose potassium iodate, so that this method may be employed in all mixtures of hypoiodites, iodates, and iodides. In one determination by this method 97 per cent of the iodine originally used was liberated on the addition of the soda-water. We may therefore conclude that when potash acts upon iodine-water there is practically no iodate formed.

As Schönbein pointed out, the solutions are very unstable. I have made a number of experiments upon the rate at which the change occurs, estimating this by the diminution in bleaching power. I find that the presence of excess of alkali makes the solution more stable; but even then a solution loses half its bleaching power on standing, in the dark, for four hours. In twenty-four hours 75 per cent of the bleaching power goes. If a much smaller amount of alkali is used half the bleaching power goes in an hour. On heating the solutions they alter very rapidly, and every bleaching liquid of this kind which I have prepared loses its bleaching power entirely if boiled for three or four minutes. As Schönbein assumed, this loss of bleaching power is doubtless due to a change of the hypoiodite into iodide and iodate,—



As mentioned above, Schwicker has recently investigated the rate at which the above change occurs at the ordinary temperature with different proportions of iodine and potash present. The results do not appear to have been altogether satisfactory. But he used iodine dissolved in potassium iodide, and there is no doubt that the latter would affect the results materially. Probably better results would be obtained by the use of a solution of hypoiodite made from hypoiodous acid, which would not contain any iodide at all.

I may refer here to the fact that whether the liquid contains iodide and hypoiodite, or iodide and iodate, the addition of an acid at once liberates the whole of the iodine; in the one case hypoiodous and hydriodic acids are liberated, which at once decompose each other—



in the other, hydriodic and iodic acids are similarly liberated, and in the exact amounts needed to decompose each other—



I have made similar bleaching solutions by using lime-water and baryta-water with aqueous iodine, and in nearly all respects these resemble Schönbein's solutions, there being perhaps a little difference in their stability in favour of the sodium and potassium compounds. They are all decomposed on boiling.

I have further found that, by using a little very finely-divided (preferably precipitated) iodine with the aqueous iodine, and then adding the alkali, very much stronger solutions may be prepared. A solution made in this way bleaches large quantities of indigo, and gives further reactions which add very strongly to the evidence that these solutions contain hypoiodites. Thus they give a black precipitate (on standing) with a cobalt solution; an immediate dark brown precipitate with a solution of a manganous salt; and with lead salts a precipitate which manifestly contains a considerable amount of the brown peroxide of lead. Also these strong solutions give an immediate and copious evolution of oxygen with hydrogen dioxide. In these reactions the solution acts exactly as the corresponding hypochlorites and hypobromites do. The dilute solutions made with aqueous iodine naturally do not give these reactions so satisfactorily unless large quantities are used. On the other hand, the stronger solutions would not be so suitable for the quantitative experiments as the more dilute ones.

The solution made with iodine-water and not too much alkali gives with nitrate of silver a precipitate which is quite distinct from the ordinary precipitated hydrate of silver, having a sort of dark buff colour. Of course the precipitate must contain silver iodide and probably also some hydrate, as the original liquid must of necessity be somewhat alkaline; but it probably also contains some silver hypoiodite. If the liquid is poured off or filtered off from the precipitate, it is found to have completely lost its bleaching power. On the other hand, if the precipitate is treated with a dilute acid, part of it dissolves up, leaving the yellow iodide of silver, and at the same time the solution acquires bleaching properties, though not to anything like the extent that would correspond to a complete transformation of the hypoiodite into a silver salt, and then to hypoiodous acid. In the two transformations a large amount of the hypoiodite is evidently decomposed.

I have already mentioned that Schönbein was greatly puzzled to account for his bleaching solutions giving a deep blue colour with starch alone. Lunge and Schoch in their paper suggested that some iodine probably existed in the liquid in combination with potassium iodide. But a much more reasonable explanation had already been supplied by the experiments of E. Lenssen and J. Löwenthal (*Journ. für Praktische Chemie*, 1862, p. 245), who found that sodium iodide and hypoiodite decompose each other, liberating iodine, and that the amount of alkali required to react with free iodine was greater when potassium iodide was present than when there was no iodide. They practically stated that the reaction—



is a balanced one, and that the addition of potassium iodide reverses the action, which now produces potash and free iodine.

It follows that the amount of alkali required to complete the above reaction must be greater than that represented by the equation. I have added varying amounts of a standard solution of soda to the same amount of aqueous iodine. With one equivalent of alkali to one of iodine the solution is distinctly yellow, and gives a deep blue colour with starch; with two equivalents of alkali the liquid is a very pale yellow, and the colour with starch is much less intense; with three equivalents the liquid appears colourless, and gives only a slight colour with starch, so that apparently the reaction is all but complete, and with four equivalents it is quite complete.

It is probable that the character of this reaction has something to do with the comparative failure to obtain bleaching solutions when using iodine dissolved in potassium iodide. It may also help to explain the fact that in the action of ozone upon potassium iodide the development of free iodine may proceed to quite a remarkable extent, considering that its liberation must be accompanied by the formation of an equivalent amount

of potash. It is clear, however, that, as there is always a very large excess of potassium iodide present, this must tend to prevent the formation of any but the smallest amount of hypoiodite.

In 1882 the paper by Lunge and Schoch on Calcium Hypoiodite appeared. The authors criticised Schönbein's work at some length. They objected to the importance which Schönbein appeared to attach to the fact that his solutions gave an evolution of oxygen with hydrogen peroxide, pointing out that a mixture of potassium iodide and iodate does the same thing. There is a certain amount of weight in this objection, but not much. It is quite true that a mixture of iodide and iodate does evolve oxygen with hydrogen dioxide, but only either on standing or when gently warmed; whereas, as I have already pointed out, the stronger hypoiodite solutions which I have prepared give an immediate violent effervescence on the addition of the peroxide. Schönbein's dilute solutions certainly do not give oxygen anything like so rapidly as these stronger ones, but still much more rapidly than a mixture of potassium iodide and iodate. I still consider, with Schönbein, that the immediate evolution of oxygen with hydrogen peroxide is a valuable indication that these solutions contain hypoiodites.

Lunge and Schoch prepared their "hypoiodite of calcium" by rubbing together for some time iodine with a large excess of lime and a comparatively small amount of water, allowing to stand for some hours, and then diluting with water. They thus obtained a solution which apparently resembled Schönbein's solutions in many respects, but gave "with cobaltous nitrate a green precipitate—no black peroxide." It bleached cochineal, logwood, and indigo carmine, just as Schönbein's solutions do.

The authors attempted to estimate the bleaching strength of the solution by means of a standard solution of indigo carmine, standardised against a dilute solution of bleaching powder, the strength of which was estimated by means of a standard solution of sodium arsenite. But they could not succeed in measuring the bleaching power of their iodine-lime solution directly, because towards the end the decolourisation was so extraordinarily slow. They therefore added an excess of indigo solution, and allowed to stand for fifteen minutes; then excess of bleaching powder solution was added, and this excess finally brought back by a drop or two of sodium arsenite solution. In this way they estimated that in their solution 14.6 per cent of the total iodine present existed as "bleaching iodine." They further stated that the solution, kept in the dark, gradually lost its bleaching power, but that only 76 per cent of the bleaching action had disappeared at the end of twenty-three days. They also tried the effect of heating the solution, and found, on one occasion, that when boiled for one hour 52 per cent of the bleaching power had disappeared. In another experiment a sample was boiled for seven hours, and then only lost 53 per cent of its bleaching power!

It is evident that there are some irreconcilable discrepancies between these results and mine. In the first place, I never found any difficulty in estimating the bleaching power of a solution directly, except in the case where the bleaching is not permanent. Secondly, my solutions gave black precipitates with cobalt; and, in the next place, every bleaching solution that I have made is decomposed completely by boiling for, at most, four minutes. Judging from the analogous bodies, hypochlorites and hypobromites, and from the instability of hypoiodites on merely keeping them in the dark, it is inherently highly improbable that any hypoiodite could stand being boiled for seven hours! I have prepared what I should call calcium hypoiodite by adding lime-water to aqueous iodine, and it decomposes completely when boiled for three minutes. Whether the complicated method adopted by Lunge and Schoch for estimating the bleaching action has anything to do with these discrepancies I am not prepared to say, but it seems quite

plain that if Schönbein's solutions consist of hypoiodites, then Lunge and Schoch's solution does not. I have tried to repeat Lunge and Schoch's experiments, following their directions, and have obtained a bleaching liquid which acts practically like Schönbein's solutions; that is, there is little or no difficulty in estimating the bleaching power directly, and it loses its bleaching power completely when boiled for a few minutes. If, also, as I should recommend, the iodine and lime are rubbed together with water, and then diluted *immediately*, instead of, as they recommend, allowing the mixture to stand for several hours, a solution is obtained three or four times as strong, which gives a dark brown precipitate with cobalt; but this also is decomposed completely when boiled for a few minutes. It also gradually decomposes when kept in the dark, and a sample tested on one occasion, after being left for three days, had lost entirely its bleaching power.

Since the appearance of Lunge and Schoch's paper there have been occasional references to hypoiodites in other papers. Thus C. Lonnes (*Zeit. Anal. Chem.*, xxxv., 409-436) has pointed out that the conversion of iodine into an iodide and an iodate by an alkali is not immediately complete, part remaining uncombined, and part being converted into hypoiodite, and that the hypoiodite has greater stability in presence of excess of alkali.

Chattaway (*Chem. Soc. Journ.*, lxi., p. 1572) has stated that in several of the decompositions which the so-called "nitrogen iodide" undergoes, hypoiodites are produced.

Quite recently (*Proc. Roy. Soc. Edin.*, xxi., 235) Dr. J. Walker and S. A. Kay, B.Sc., have published a paper on the so-called "Magnesium Hypoiodite," a brown substance formed by the union of magnesia, either wet or dry, with free iodine, and which has sometimes been supposed to be magnesium hypoiodite. They conclude, however, that it is simply a case of absorption of iodine, without any chemical combination. They find that this brown precipitate is produced when potash is added to a solution of iodine in potassium iodide until the iodine just disappears, and then a solution of magnesium sulphate is added, magnesium hydrate being precipitated, and iodine manifestly liberated. They have concluded from this that, as pointed out above, the reaction between iodine, potash, potassium iodide, and water, is a balanced one.

(To be continued).

THE PREPARATION OF ZINC ETHYL.

By ARTHUR LACHMAN.

Of all the methods heretofore proposed for the preparation of zinc alkyls, the Gladstone-Tribe copper-zinc couple (*J. Chem. Soc.*, 1879, 570), gives by far the best results. As originally given, however, the directions are open to two practical objections. First of all, it is not always an easy matter to secure the zinc filings of necessary fineness. And secondly, while the careful fusion of the filings with the reduced copper offers no difficulties when carried out with 10-grm. lots, as usually taken by the authors, it is practically impossible to prevent larger quantities from melting completely and forming a worthless solid ingot on cooling.

A form of zinc available in every laboratory is zinc dust. On removing the coating of oxide by heating in hydrogen for a short while, it was found that 50 grms. each of the metal and ethyl iodide reacted within ninety minutes, and gave 14 grms. of zinc ethyl (72 per cent of the theoretical yield). When this metal is coupled with copper, by reducing an intimate mixture of zinc dust and finely powdered copper oxide, the time of reaction is shortened to thirty minutes, and the yield increased to nearly 90 per cent. Different varieties of zinc dust give

different yields (ranging from 70 per cent upward); this probably finds its explanation in the phenomena discussed by H. Wislicenus (*J. prakt. Chem.* (2), liv., 18 ff). The actual quantities of materials taken also exercise an influence, the yield being somewhat smaller with larger quantities (100 grms. or more). I have not attempted to ascertain the cause of this.

The details of the process are extremely simple. Zinc dust and copper oxide are mixed in the ratio of 100 to 12, loosely filled into a combustion tube, and a current of hydrogen passed over the heated mixture for about twenty minutes. In order to prevent the condensation of moisture in the tube, with the consequent caking of the mass, it is best to heat the tube along its whole length at once. A dull red heat seems to give the best results; the nearer the melting-point of zinc the better, I presume. If the temperature be kept too low, the metals do not alloy well, and the results approach those given by zinc dust alone. I have usually employed equal quantities of ethyl iodide and metal couple, sometimes less metal; perhaps the yield might be increased by excess thereof. For further directions the article of Gladstone and Tribe had best be consulted. In place of the oil-bath for heating the zinc ethiodide, the Babo air-baths may be substituted, as being cleaner and more expeditious; if a small flame be placed under them at first, there is no danger of breaking the flask. By employing two sets of apparatus, and working up about 100 grms. at a time, the process may easily be made continuous, and several hundred grms. of zinc ethyl prepared in a single day. Mr. F. M. Tschirner, who has manufactured nearly 300 grms. for me, found no difficulty in obtaining 150 grms. in an afternoon.

It is customary to preserve zinc ethyl in sealed tubes. This is awkward where small and varying quantities are frequently needed. Small Erlenmeyer flasks, with perfectly straight sides beginning at the very mouth, greatly facilitate the operation of pouring out the liquid, and when well corked form perfectly safe receptacles. Instead of requiring an assistant to keep a stream of carbon dioxide upon the mouth of the flask when transferring zinc ethyl, it is much more convenient to conduct all such manipulations under a small, inverted funnel, through which the gas is rapidly passing.

It had been my intention to compare the above described modification of the copper-zinc couple with the original, chiefly with respect to its reducing powers, but the recent article of H. Wislicenus (*loc. cit.*) has made this unnecessary. Qualitative tests show that the new form reacts in much the same manner with alkyl and alkylene haloids as the older. I have attempted to use ethyl bromide in place of the expensive iodide, but without encouraging results. Methyl iodide reacts as readily as the ethyl compound, though the yield of zinc methyl was not determined.—*American Chemical Journal*, xix., No. 5.

A SIMPLE TEST FOR THE HALOGENS IN ORGANIC HALIDES.

By J. H. KASTLE and W. A. BEATTY.

IN connection with some work on the displacement of bromine and iodine from their organic compounds, we had frequent occasion to test for the halogens in their organic derivatives, and, while a great many methods for conducting such test were to be found in the literature, few, if any of these, could be conducted rapidly; and many involved the use of rather large amounts of substances, such as lime, soda-lime, &c., which are not readily obtained free from chlorine. It is unnecessary in this connection to enumerate the many tests which have been proposed for these elements in their organic compounds; most of them are already familiar to chemists,

and nearly all of them are described in Beilstein's "Handbuch" and in Allen's "Commercial Organic Analysis."

Appreciating the need therefore of a simpler and rapid test whereby chlorine, bromine, and iodine could be recognised with certainty in their most stable and volatile organic combinations, it occurred to one of us (Kastle) to heat such compounds with a mixture of silver and copper nitrates, thereby oxidising the organic matter and holding back any halogen present by means of silver. On experiment it was found that excellent results could be obtained with this mixture, which can always be obtained free from any traces of chlorine, bromine, or iodine, by dissolving the metals in pure nitric acid and evaporating to crystallisation. In the case of non-volatile substances the test is most easily conducted as follows: A small quantity of a substance to be tested, usually about 0.1 grm., is placed in the test-tube, along with about 0.5 grm. of the mixed nitrates and a few drops of water. The tube is then gradually heated in the flame of the Bunsen burner until the nitrates are completely decomposed; the temperature never being allowed to rise higher than low red heat. The tube, in which some reduced copper is often seen, is allowed to cool, a little water and dilute sulphuric acid poured on the contents of the tube and a few pieces of metallic zinc added. After five or ten minutes, during which time any halogen compound of silver is reduced, the contents of the tube are filtered and a solution of silver nitrate added to the filtrate, together with a little dilute nitrate acid, when the chloride, bromide, or iodide of silver will be precipitated, if either of these halogens was present in the substance tested.

Excellent tests for chlorine, bromine, and iodine were obtained in this way in the following substances: Dibromnaphthalene, eosine, benzene dichlorosulphonamide, *p*-brombenzene-sulphonamide, tribromphenol, potassium *p*-brombenzenesulphonate, monobromacetanilide, soxodol (Merck), barium *p*-iodobenzenesulphonate. With very volatile substances such as chloroform, it was found necessary to conduct the test in a tube about six inches long by one-quarter inch internal diameter, closed at one end, and bent twice at right angles at equal distances along the tube, so as to form a tube having somewhat the shape of the letter S. In making the test about $\frac{1}{2}$ c.c. of the volatile compound to be tested was introduced into the closed end of the tube, and the tube so clamped as to give it open end an upward slant. About 0.5 grm. of the dry mixed nitrates was then placed in the bend of the tube farthest removed from the closed end; after which the parts of the tube containing the substance to be tested and the nitrates were heated alternately, the former portion very gently, so as not to drive out the volatile substance before it could be oxidised by the decomposing nitrates. The heating is continued until all the nitrates are decomposed, and all of the substance to be treated volatilised, at the end of which time the tube is broken and the fragments, containing the oxides of copper and silver, placed in a test-tube with a small quantity of water and sulphuric acid and a few small pieces of zinc. The test is then completed in the manner described in the above. The following substances were tested in the closed S-shaped tubes with excellent results in every case:—

Chloroform, isopropyl bromide, butyl bromide, ethyl chlorcarbonate, propyl bromide, propyl iodide, bromoform, ethylidene chloride, propyl chloride, monobrombenzene, dibrombenzene, ethylene bromide, monochloroacetic acid, benzoyl chloride, dibromsuccinic acid.

It is believed that this test as applied both to volatile and to non-volatile organic halides has in it many points of advantage over those which have been proposed for this purpose; in the first place the test is rapid and certain in its result, and by means of it we can judge not only of the presence or absence of the halogens, but it can also be determined at a glance whether it is chlorine, bromine, or iodine that is present in the organic compound. Secondly, only very small quantities of sub-

stances are required to make the tests, and the few simple reagents needed may be obtained free from any traces of halogen without the least difficulty.—*American Chemical Journal*, xix., No. 5.

A NOTE ON THE EFFECT OF PRESSURE UPON THE SERIES IN THE SPECTRUM OF AN ELEMENT.

By J. S. AMES and W. J. HUMPHREYS.

It has been known for many years that the spectra of certain elements, notably the alkalis and the alkaline earths, contained series of lines, which obeyed a mathematical law like that giving the distribution of lines in the spectrum of hydrogen. These series have been thoroughly studied by Kayser and Runge, who have classified them according to their physical characteristics as principal, first subordinate, second subordinate. Only a few elements, lithium, sodium, potassium, have in their spectra all three series; while the last two series, the subordinate ones so-called, are common to some ten others; the wave-lengths being different in the spectra of the different elements, the physical properties being the same.

While the shift of the lines in the spectra of the elements was under investigation, it seemed important to study in particular the effect of pressure upon the series of the various elements. To this end, photographs were taken of the arc-spectra of all elements which give series, both at ordinary pressure and at increased pressure; and the shifts were carefully measured of as many of the lines as possible. In certain cases eye-observations were also made. The results for each element may be thus briefly stated:—

1. The lines of any one series of a particular element are shifted alike, *i. e.*, according to the same law, which may be written:—

$$\Delta\lambda = \lambda\beta(p_1 - p_0)$$

where λ is the wave-length, $\Delta\lambda$ is the shift produced by an increase of pressure $p_1 - p_0$, β is a constant for any one series of a definite element.

2. The constant β is different for the different series of the same element, the change being such that, *very nearly*, β for the principal series is one-half β for the first subordinate and one-quarter that of the second subordinate.

3. The constant β is different for the same series of different elements. Special attention is called to this fact in another note in this Circular. One apparent irregularity which demands attention is the fact that, approximately, the value of β for similar elements (*e. g.*, zinc, cadmium, mercury) varies as the cube-root of the atomic weight.

No satisfactory theory has been advanced to account for these shifts of the spectrum lines when the arc is under pressure. There is every evidence that it is not due to a temperature effect of any ordinary kind. It would certainly be expected that the outer envelope of an arc would be at a much lower temperature than the core, and that different electric currents might cause different temperatures in the arc; yet no shift due to these variations has been observed. It is our intention to observe the arc under these conditions with a Michelson refractometer as soon as possible, and so to learn what actually occurs. Again, the temperature of the arc should be much greater than that of a Bunsen flame which is being fed with sodium; but the difference in the wave-length of D_2 under the two conditions is not perceptible with a 21 ft. concave grating, 15,000 lines to the inch; *i. e.*, a shift, if any, must be less than 0.002 of an Angström unit. The words "temperature of the arc" are used with considerable hesitation, because so little is known as to the mean condition of the molecules of the vapour which are producing the light.

One can easily understand, however, that, if the pressure

on a gas is increased, the number of collisions per second must increase; and it is not impossible that this increased internal energy of a molecule, as thus produced, is the immediate cause of a change in the actual size of the molecule. The extent of this change would depend upon the looseness of construction of the molecule, apparently; and this quantity is measured to a certain degree by the coefficient of expansion of the element in the solid form. Therefore, it might be expected that the measured shift would vary in the same direction as the coefficient of expansion of the solid. This is actually the case, with no exception. Again, since on any theory of emission of waves the wave length varies directly as the linear dimension of the portion of matter producing the waves, it would be expected that the measured shift would vary directly as the coefficient of linear expansion, which is found to be the case. If α is this coefficient, it may be stated as an experimental law that, for different elements $\beta = c\alpha$ where c is a constant, which in some way connects the ordinary changes in size of a solid due to temperature changes with the hypothetical changes in the size of the molecules of the vapour due to pressure changes.

It is not difficult to see that one would expect, as consequences of the above ideas, that the shift would be proportional to the total increase of pressure, regardless of its mode of production; and that it should also vary directly as the wave-length in any one series or group of lines, for in such a case the longer waves indicate greater linear dimensions of the vibrating segments, if the term may be used. These are observed phenomena, as is stated above.

The fact that the shift characteristic of a principal series is less than that of the first subordinate, and this in turn less than that of the second subordinate would be expected, in accordance with these ideas, if the molecules producing the principal series were of a simpler structure than those producing the first subsidiary; and if the molecules producing the second subsidiary were the most complex of all. For, since the shift of a series depends upon the looseness of the molecular structure, it would be expected that, if these molecules split up in any way, the fragments would be more stable and firm than the original molecules, and therefore the shift of the original molecules would be greater than that of the fragments. It is difficult, however, to see any reason why the shift of the different series should vary according to any simple law, or why the shift of the same series of different elements should be in accordance with any formula so simple as that of the cube root of the atomic weight. If it could be assumed that the shift was proportional to the linear dimensions of the segment producing the waves, most interesting deductions might be drawn; but there seems to be no justification for the assumption.—*Johns Hopkins University Circular*, xvi., No. 130.

SURFACE TENSION OF WATER AND OF DILUTE AQUEOUS SOLUTIONS.

By N. ERNEST DORSEY.

DURING the past year I have been endeavouring to determine the surface tension of dilute aqueous solutions by means of the method of ripples. All work previously done on the surface tension of solutions has been on solutions of about one-half normal concentration, or greater, and most of the observers have deduced the surface tension from the measured rise of the solution in capillary tubes.

For at least two reasons the method of capillary tubes is open to serious objections. First, the height a liquid rises in a tube depends upon the angle between the wall of the tube and the surface of the liquid where it meets the tube. This contact angle can not possibly be measured, since the surface of the liquid lies entirely on one side of the point where we wish to know its inclination; and as

we can measure the inclination of a finite surface only every measured value of the contact angle must be too large.

The second objection is that probably the surface tension of the solution-glass surface, as well as that of the solution-air surface, varies with the concentration of the solution. If such is the case the surface tension found will depend upon two changes which can not be readily separated, and which render the interpretation of the results difficult.

For these reasons I decided to use the method of ripples, which was first successfully used by Lord Rayleigh, although with his arrangement of apparatus individual observations differ by about 2 per cent. After trying many plans one was finally adopted that gives individual results that agree to about $\frac{1}{2}$ per cent; and the average departure of single observations from the mean of several seldom exceeds $\frac{1}{2}$ per cent.

The waves were generated by a fork whose frequency was often determined and was always near 62.87 double vibrations per second. The water and solutions were contained in a porcelain tray 1 by 12 by 14 inches. The wave length was measured by means of a telescope mounted on a dividing engine, whose screw had a pitch of 1.0328 m.m. The waves were visible under ordinary conditions, but were observed by Foucault's method for rendering visible small vibrations in plane or spherical surfaces.

The water used was especially distilled by Mr. W. T. Mather from chromic acid and alkaline potassium permanganate, and was condensed in a block tin condenser; it was the kind used by him for his electrolytic work. The salts were obtained from Bimer and Amend and were said to be chemically pure.

With this apparatus I have determined the surface tension of water and of solutions of sodium chloride, potassium chloride, sodium carbonate, potassium carbonate, and zinc sulphate, of concentrations varying from 0.05 normal to normal.

The value found for water is $T = 75.98$ dynes per centimetre at 0°C ., while *Sentis Jour. de Phys.* (3) vi., 183, 1897) working by an entirely different method found $T = 76.09$ at 0°C ., which differs from the other by only 0.14 per cent. These values agree very well with the values given by Lord Rayleigh, Hall, Volkmann, and others, but are much lower than Quincke's value.

It was found that the surface tensions of dilute aqueous solutions are linear functions of the concentration; so that we may write $T_s = T_w + kC$, where T_s = surface tension of the solution, T_w = surface tension of water at the same temperature, k is a constant, C is the concentration in grm. molecules per litre. Below is a table showing the value of k as determined by different observers.

	Dorsey.	Volkmann.	Quincke.	Rother.
NaCl $k =$	1.53	1.59	1.57	1.38
KCl	2.23	1.41	1.57	1.47
$\frac{1}{2}$ Na ₂ CO ₃	2.00	0.987	1.57	—
$\frac{1}{2}$ K ₂ CO ₃	1.77	1.78	1.57	—
ZnSO ₄	1.86	—	—	—

Volkmann found that the curve for Na₂CO₃ at great dilution becomes steeper than the one for K₂CO₃, which agrees with my results. Quincke's value, 1.57, does not agree with his results except for KCl and NaCl. I cannot account for the very high value I found for KCl, but it must be borne in mind that the values given above are not fairly comparable, since my values are for solutions generally less concentrated than $\frac{1}{2}$ normal, while the others are found for solutions of greater concentration.—*Johns Hopkins University Circular*, xvi., No. 130.

A Menthoglycol.—Ph. Barbier and G. Lenz.—In addition to isopulegol and menthoglycol, there is formed during the action of dilute sulphuric acid upon citronallol, a small quantity of C₂₀H₃₄O, a body boiling at 185° under the pressure of 10 m.m.—*Comptes Rendus*, cxxiv., No. 23.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 23, June 8, 1897.

Correspondence.—The Secretary read a letter announcing the dispatch of 25,000 francs derived from a subscription gathered in Russia as a contribution to the fund for the erection of a monument to Lavoisier.

Properties of Simple Kathodic Rays. Relations with Simple Electric Radiations.—H. Deslandres.—The author's experiments, and in particular an experiment with the Tesla-d'Arsonval arrangement, lead to the following conclusion:—The simple kathodic rays correspond to the simple electric oscillations. Further, the Crookes tube, completed by arrangements described in the author's paper, constitutes an apparatus capable of furnishing rapid and valuable indications on the electrification of conductors submitted to high tensions.

Atomic Weight of Cerium.—M. Wyrouboff and A. Verneuil.—On operating with products strictly pure, and employing methods as free as possible from errors, cerium, of whatever origin, shows an atomic weight very close upon 92.7. Considering the indirect character of the method employed, this figure can only be considered as approximative to about 0.2–0.3.

Heat Liberated on the addition of Bromine to some Non-saturated Substances.—W. Louguine and Jas. Kablukow.—The addition-heats of Br to allylic alcohol and its derivatives vary little among themselves. For allyl chloride and bromide they are almost equal. The substitution of phenyl (C₆H₅) for hydrogen in allylic alcohol diminishes notably the addition-heat of bromine.

Combinations of Phenylhydrazine with Metallic Bromides.—J. Moitessier.—Not suitable for abridgment.

Chemical Study on the Culture of the Cattleya.—Alex Hébert and G. Truffaut.—The generated bulbs contain a diminished proportion of dry matter, of organic and nitrogenous matter, and of ash; the decrease falling principally upon the potassa, lime, magnesia, and phosphoric acid.

Examination of Aluminium Utensils.—M. Balland.—The aluminium utensils must contain 99 to 99.5 of pure aluminium. In alloys of copper and aluminium, the copper must not exceed 2 to 3 per cent. The vessels, &c., must not be cleansed with soda.

Bulletin de la Société Chimique de Paris.

Series 3, Vol. xvii.-xviii., No. 10. May 20, 1897.

Action of Dilute Nitric, Sulphuric, Hydrochloric, and Phosphoric Acids on Nitrates in the Presence of Ether.—C. Tanret.—When two liquids, insoluble in each other, one of which has in solution a body equally soluble in the other, are shaken up together, these two liquids will divide the soluble body between them in such a manner that the quantity as dissolved in given volumes of each will always have a constant relation one to the other. Having occasion to apply this method to the detection of small quantities of nitric acid in a liquid rich in nitrate of ammonia, the author obtained some quite unexpected results. The acid dissolved in ether as an organic acid would have done, then, after agitation with water, passed to a great extent into the latter. Nitric acid therefore possesses two coefficients, according to whether nitrate of ammonia is present or not. This research was the outcome of this observation, and the author maintains that the action of nitrates on the coefficient of "division"

of the nitric acid is explained if we admit that, in dissolving in nitric acid, the neutral nitrate becomes an acid nitrate. Now, water does not completely decompose acid salts into free acid and neutral salts, because these two tend to re-combine and re-form the original salt. It follows, then, that in adding either an excess of acid with regard to the amount of neutral salt present, or an excess of neutral salt with regard to the acid present, the tendency is to augment the stability of the acid salt. This latter case is precisely the result obtained in these experiments.

Formation of Metallic Sulphides by Mechanical Agencies.—L. Franck.—This paper contains an account of several simple experiments, which prove the combination of two bodies by mechanical influence and mutual contact, by the friction of the two bodies. Small quantities of sulphides have been obtained by rubbing flowers of sulphur, and the powder of certain metals, —such as iron, copper, aluminium, &c.,—between two sheets of paper.

Action of Chloride of Chloracetyl on some Aromatic Hydrocarbons, in the presence of Chloride of Aluminium.—A. Collet.—In a previous note the author described the action of various chlorides of acid halogens on benzene in the presence of chloride of aluminium; he now describes the products obtained with chloride of chloracetyl in a similar manner.

On some Derivatives of Anise-aldehyde.—A. Reychler.—Not suitable for abstraction.

Contribution to the Study of Coumarin.—A. Reychler.—The author finds that by using pure by-products, and working at a sufficiently high temperature, the Perkin reaction does not appear to need the presence of any great proportion of alkaline salts.

Products of the Action of Benzhydrol-diamide and Tetramethyl, on Para- and Meta-sulphanilic Acids.—M. Soais.—Not suitable for abstraction.

On a New Method of Extracting the Perfume from Flowers.—J. Passy.—The author soaks the flowers in water, without killing them; the scent goes into the water, as it does into air; the water is then treated with ether, which collects the scent. This renders maceration unnecessary.

Study of Chlorophyll.—J. Stoklasa.—The author finds that the cellular germ does not form—not only without phosphorus but also without iron—if the chlorophyll contains only phosphorus.

Schiff Reaction applied to Acid Fuschine.—L. Lefevre.—The author finds that M. Cazeneuve's conclusion, that fuschine decolourised by SO₂ would not regain its colour when treated with aldehyds, is not correct. The success of the reaction depends entirely on the quantities used.

Bulletin des Travaux de la Société de Pharmacie de Bordeaux. May, 1897.

New Method of Testing for Biliary Calculus.—G. Denigès.—Not suitable for abstraction.

Experiments on Commercial Albumen.—P. Carles.—The adoption of albumen as a clarifier by several industries has brought a number of different brands on the market; some can be used with every confidence, but unfortunately, with a view to cheapen its production, there are also brands carelessly made, and, what is worse, adulterated. Some samples have been found to contain from 12 to 25 per cent of insoluble coagulated matter, having no clarifying power whatever. Gum, dextrine, and gelatin are also used as adulterants. To examine the purity of albumen, 2 grms. should be made into a paste with distilled water, then more water gradually added, up to 200 c.c.; if the albumen be free from coagulated particles this solution will be translucent. To 100 c.c. of

this solution add 35 c.c. of a 1 per cent solution of tannin, then 0.2 grm. of powdered bitartrate of potash. Shake well and filter; to one part of the filtrate add a few drops of a solution of five parts per thousand of grenatine, and to the other a few drops of the tannin solution above mentioned. If there is no change the albumen is pure; if the grenatine gives a precipitate it shows that there is tannin in excess, and, therefore, that the albumen is either adulterated with some inert body or has been over-heated in making; if, however, the tannin gives a precipitate in the last tube, it proves the presence of gelatin in the sample, as gelatin will precipitate, weight for weight, four times as much tannin as dried albumen can.

Phenomenon of Oxidation produced by Different Milks.—R. Dupouy.—Not suitable for abstraction.

New Form of Oven for Desiccation and Sterilisation.—M. Soulard.—The large number of different desiccating ovens now used in laboratories show that perfection is very difficult to attain. Physical science teaches us that the rapidity of desiccation depends on the temperature, on the degree of saturation of the surrounding atmosphere, on the renewing of this atmosphere, and on the amount of surface for evaporation. Most ovens will not allow of the conditions being at their best. The oven made by the author seems to conform to the state of things required. It is made of sheet copper, with double walls, and so ingeniously put together that the inner chamber is heated directly by the flame, while at the same time a current of hot fresh air enters at one side near the bottom, and by means of alternate trays circulates—or rather zigzags—throughout the interior until it reaches the top, where it escapes. The temperature can be so regulated that the oven can be used for evaporations and desiccations, or it can be raised to and maintained at 160° or 200° for sterilisations.

Société de l'Industrie Minérale, Comptes Rendus Mensuels.
April, 1897.

Electrolysis of Solid Bodies.—M. Mayençon.—For these experiments a bichromate battery of six cells is sufficient, and the whole apparatus necessary is of a very simple character—such as an agate mortar, a platinum crucible, one or two spatulas, a glass funnel, &c. The action of the current is shown first on a few simple bodies capable of forming acids; for instance, carbon is transformed by the oxygen of water into carbonic acid, when it is employed as the anode, the cathode being of platinum. To show this a small quantity of baryta water is poured into a test-tube, and a few drops of chloride of barium added; when the current is passed through as described, a white precipitate of carbonate of baryta is formed, soluble in acids. In a like manner sulphur forms sulphuric acid, and then sulphate of barium when moistened with chloride of barium. As a final experiment on insoluble simple bodies, phosphorus was transformed into phosphoric acid, by passing the current through small pieces of phosphorus moistened with nitro-molybdate of ammonia, when the yellow phospho-molybdate of ammonia—insoluble in acids and soluble in ammonia—was formed.

The decomposition of a silicate by means of the electric current is also performed, but it is not easy to show the presence of silica by absolutely characteristic reactions. Orthose gives on one pole silica, at the other potash, and garnierite in a similar manner gives metallic nickel. Further experiments are being made, and will shortly be communicated.

Bulletin de la Société d'Encouragement pour l'Industrie Nationale. Series 5, Vol. ii., No. 5. May, 1897.

Estimation of the Oxidation of Oils.—W. Bishop.—This is a report to the Society by M. Bérard, on the work done by M. Bishop on this subject. This work was inspired by an experiment made by Chevreul in 1856, in which he found that linseed oil mixed with manganese

absorbed ten times more oxygen than the pure oil itself. M. Bishop, however, uses as drying agent a kind of soap made by mixing oxide of manganese with resin. He dissolves this resin in the oil to be tested in the proportion of 0.2 grm. of the former to 10 grms. of the oil. Then, to facilitate the access of air, he adds 1 grm. of a light silicate, obtained by precipitation and calcination. The desiccation is then allowed to proceed. The table of the order of oxidation confirms what has already been shown by M. Livache with regard to this order. M. Bishop obtained for linseed oil an absorptive value of oxygen of 14 per cent in twenty-four hours. He has successfully applied his method to the comparison of different commercial oils and the recognition of possible mixtures which could be made.

Composition of Clays.—Georges Vogt.—A very long paper, not suitable for abstraction.

Researches on the Colouration of Glass by the Direct Penetration of Metals or Metallic Salts.—Leon Lémal.—Reprinted from the *Comptes Rendus*, May 17, 1897.

On Solutions of Acetylene and their Explosive Properties.—MM. Berthelot and Vieille.—From the *Comptes Rendus*, May 10, 1897.

THE DAVY FARADAY RESEARCH LABORATORY OF THE ROYAL INSTITUTION.

Directors:
The Right Hon. LORD RAYLEIGH, M.A., D.C.L., LL.D., F.R.S.
Professor DEWAR, M.A., LL.D., F.R.S.
Superintendent of the Laboratory:
Dr. ALEXANDER SCOTT, M.A., D.Sc.

This Laboratory, which has been founded by Dr. LUDWIG MOND, F.R.S., as a Memorial of Davy and Faraday "for the purpose of promoting original research in Pure and Physical Chemistry," is now open. The next Term begins on the 4th of October, 1897.

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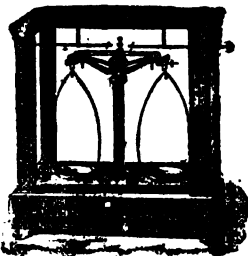
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VOL. LXXVI., No. 1964.

DIAMONDS.*

By WILLIAM CROOKES, F.R.S., M.R.I.

(Concluded from p. 15).

The Mechanism of the Diamantiferous Pipes.

How the great diamond pipes originally came into existence is not difficult to understand, in the light of the foregoing facts. They certainly were not burst through in the ordinary manner of volcanic eruption; the surrounding and enclosing walls show no signs of igneous action, and are not shattered nor broken even when touching the "blue ground." These pipes after they were pierced were filled from below, and the diamonds formed at some previous epoch too remote to imagine were erupted with a mud volcano, together with all kinds of *débris* eroded from the adjacent rocks. The direction of flow is seen in the upturned edges of some of the strata of shale in the walls, although I was unable at great depths to see any upturning in most parts of the walls of the De Beers mine.

Let me again refer you to the picture of the section through the Kimberley mine. There are many such pipes in the immediate neighbourhood. It may be that each volcanic pipe is the vent for its own special laboratory—a laboratory buried at vastly greater depths than we have reached or are likely to reach—where the temperature is comparable with that of the electric furnace, where the pressure is fiercer than in our puny laboratories and the melting-point higher, where no oxygen is present, and where masses of carbon-saturated iron have taken centuries, perhaps thousands of years, to cool to the solidifying point. Such being the conditions, the wonder is, not that diamonds are found as big as one's fist, but that they are not found as big as one's head. The chemist arduously manufactures infinitesimal diamonds, valueless as ornamental gems; but Nature, with unlimited temperature, inconceivable pressure, and gigantic material, to say nothing of measureless time, produces without stint the dazzling, radiant, beautiful crystals I am enabled to show you to-night.

The ferric origin of the diamond is corroborated in many ways. The country round Kimberley is remarkable for its ferruginous character, and iron-saturated soil is popularly regarded as one of the indications of the near presence of diamonds. Certain artificial diamonds present the appearance of an elongated drop. From Kimberley I have with me diamonds which have exactly the appearance of drops of liquid separated in a pasty condition and crystallised on cooling. At Kimberley and in other parts of the world, diamonds have been found with little appearance of crystallisation but with rounded forms similar to those which a liquid might assume if kept in the midst of another liquid with which it would not mix. Other drops of liquid carbon retained above their melting-point for sufficient time would coalesce with adjacent drops, and on slow cooling would separate in the form of large perfect crystals. Two drops, joining after incipient crystallisation, would assume the not uncommon form of interpenetrating twin crystals. Illustrations of these forms from Kimberley are here to-night. Other modified circumstances would produce diamonds presenting a confused mass of boarty crystals, rounded and amorphous masses, or a hard black form of carbonado.

Again, diamond crystals are almost invariably perfect

on all sides. They show no irregular side or face by which they were attached to a support, as do artificial crystals of chemical salts; another proof that the diamond must have crystallised from a dense liquid.

When raised the diamond is in a state of enormous strain, as I have already shown by means of polarised light. Some diamonds exhibit cavities which the same test proves to contain gas at considerable pressure.

The ash left after burning a diamond invariably contains iron as its chief constituent; and the most common colours of diamonds, when not perfectly pellucid, show various shades of brown and yellow, from the palest "off colour" to almost black. These variations accord with the theory that the diamond has separated from molten iron, and also explains how it happens that stones from different mines, and even from different parts of the same mine, differ from each other. Along with carbon, molten iron dissolves other bodies which possess tinctorial powers. One batch of iron might contain an impurity colouring the stones blue, another lot would tend towards the formation of pink stones, another of green, and so on. Traces of cobalt, nickel, chromium, and manganese, metals present in the blue ground, might produce all these colours.

An hypothesis, however, is of little value if it only elucidates one half of a problem. Let us see how far we can follow out the ferric hypothesis to explain the volcanic pipes. In the first place we must remember these so-called volcanic vents are admittedly not filled with the eruptive rocks, scoriaceous fragments, &c., constituting the ordinary contents of volcanic ducts. At Kimberley the pipes are filled with a geological plum pudding of heterogeneous character—agreeing, however, in one particular. The appearance of shale and fragments of other rocks shows that the mélange has suffered no great heat in its present condition, and that it has been erupted from great depths by the agency of water vapour or some similar gas. How is this to be accounted for?

It must be borne in mind I start with the reasonable supposition that at a sufficient depth* there were masses of molten iron at great pressure and high temperature, holding carbon in solution, ready to crystallise out on cooling. In illustration I may cite the masses of erupted iron in Greenland. Far back in time the cooling from above caused cracks in superjacent strata through which water† found its way. On reaching the iron the water would be converted into gas, and this gas would rapidly disintegrate and erode the channels through which it passed, grooving a passage more and more vertical in the endeavour to find the quickest vent to the surface. But steam in the presence of molten or even red-hot iron rapidly attacks it, oxidises the metal and liberates large volumes of hydrogen gas, together with less quantities of hydrocarbon‡ of all kinds—liquid, gaseous, and solid. Erosion commenced by steam would be continued by the other gases, and it would be no difficult task for pipes, large as any found in South Africa, to be scored out in this manner. Sir Andrew Noble has shown that when the screw stopper of his steel cylinders in which gunpowder explodes under pressure is not absolutely perfect, gas finds its way out with a rush so overpowering as to score a wide channel in the metal; some of these stoppers and vents are on the table. To illustrate my argument Sir Andrew Noble has been kind enough to try a special experiment. Through a cylinder of granite is drilled a hole 0.2 inch diameter, the size of a small vent. This is made the stopper of an explosion chamber, in which a quantity of cordite is fired, the gases escaping through the granite vent. The pressure is about 1500 atmospheres, and the whole time of escape

* The requisite pressure of fifteen tons on the square inch would exist not many miles beneath the surface of the earth.

† There are abundant signs that a considerable portion of this part of Africa was once under water, and a fresh-water shell has been found in apparently undisturbed blue ground at Kimberley.

‡ The water sunk in wells close to the Kimberley mine is sometimes impregnated with paraffin, and Sir H. Roscoe extracted a solid hydrocarbon from the "blue ground."

* A Lecture delivered at the Royal Institution, Friday, June 11th, 1897.

is less than half a second. Notice the erosion produced by the escaping gases and by the heat of friction, which have scored out a channel over half an inch diameter and melted the granite along their course. If steel and granite are thus vulnerable at comparatively moderate gaseous pressure, is it not easy to imagine the destructive upburst of hydrogen and water-gas grooving for itself a channel in the diabase and quartzite, tearing fragments from resisting rocks, covering the country with débris, and finally, at the subsidence of the great rush, filling the self-made pipe with a water-borne magma in which rocks, minerals, iron oxide, shale, petroleum, and diamonds are churned together in a veritable witch's cauldron! As the heat abated the water vapour would gradually give place to hot water, which forced through the magma would change some of the mineral fragments into the now existing forms.

Each outbreak would form a dome-shaped hill, but the eroding agency of water and ice would plane these eminences until all traces of the original pipes were lost.

Actions, such as I have described, need not have taken place simultaneously. As there must have been many molten masses of iron with variable contents of carbon, different kinds of colouring matter, solidifying with varying degrees of rapidity, and coming in contact with water at intervals throughout long periods of geological time—so must there have been many outbursts and upheavals, giving rise to pipes containing diamonds. And these diamonds, by sparseness of distribution, crystalline character, difference of tint, purity of colour, varying hardness, brittleness and state of tension, would have impressed upon them, engraved by natural forces, the story of their origin—a story which future generations of scientific men may be able to interpret with greater precision than we can to-day.

Who knows but that at unknown depths in the earth's metallic core beneath the present pipes there are still masses of iron not yet disintegrated and oxidised by aqueous vapour,—masses containing diamonds, unbroken, and in greater profusion than they exist in the present blue ground, inasmuch as they are enclosed in the matrix itself, undiluted by the numerous rock constituents which compose the bulk of the blue ground.

Meteoritic Diamonds.

There is another diamond theory which appeals to the fancy. It is said that the diamond is a direct gift from Heaven, conveyed to earth in meteoric showers. The suggestion, I believe, was first broached by A. Meydenbauer (*CHEMICAL NEWS*, xli., p. 209, 1890), who says:—"The diamond can only be of cosmic origin, having fallen as a meteorite at later periods of the earth's formation. The available localities of the diamond contain the residues of not very compact meteoric masses, which may, perhaps, have fallen in historic ages, and which have penetrated more or less deeply, according to the more or less resistant character of the surface where they fell. Their remains are crumbling away on exposure to the air and sun, and the rain has long ago washed away all prominent masses. The enclosed diamonds have remained scattered in the river beds, while the fine light matrix has been swept away."

According to this hypothesis, the so-called volcanic pipes are simply holes bored in the solid earth by the impact of monstrous meteors—the larger masses boring the holes, while the smaller masses, disintegrating in their fall, distributed diamonds broadcast. Bizarre as such a theory may appear, I am bound to say there are many circumstances which show that the notion of the heavens raining diamonds is not impossible.

In 1846 a meteorite fell in Hungary (the "Ava meteorite") which was found to contain graphite in the cubic crystalline system. G. Rose thought this cubic graphite was produced by the transformation of a diamond. Long after this prediction was verified by Weinschenk, who found transparent crystals in the Ava meteorite. Mr. Fletcher has found in two meteoric irons

—one from Yondeggin, East Australia, and one from Crosby's Creek, United States—crystals absolutely similar to those in the Ava meteorite.

In 1886 a meteorite falling in Russia contained, besides other constituents, about 1 per cent of carbon in light grey grains, having the hardness of diamond, and burning in oxygen to carbonic acid.

Daubrée says the resemblance is manifest between the diamantiferous earth of South Africa and the Ava meteorite, of which the stony substance consists almost entirely of peridot. Peridot being the inseparable companion of meteoric iron, the presence of diamonds in the meteorites of Ava, of Yondeggin, and of Crosby's Creek, bring them close to the terrestrial diamantiferous rocks.

Hudleston maintains that the bronzite of the Kimberley blue ground is in a condition much resembling the bronzite grains of meteorites; whilst Maskelyne says that the bronzite crystals of Dutoitspan resemble closely those of the bronzite of the meteor of Breitenbach, but are less rich in crystallographic planes.

But the most striking confirmation of the meteoric theory comes from Arizona. Here, on a broad open plain, over an area about five miles diameter, were scattered one or two thousand masses of metallic iron, the fragments varying in weight from half a ton to a fraction of an ounce. There is little doubt these masses formed part of a meteoric shower, although no record exists as to when the fall took place. Curiously enough, near the centre, where most of the meteorites have been found, is a crater with raised edges, three-quarters of a mile in diameter, and about 600 feet deep, bearing exactly the appearance which would be produced had a mighty mass of iron or falling star struck the ground, scattered in all directions, and buried itself deep under the surface. Altogether ten tons of this iron have already been collected, and specimens of the Canyon Diablo meteorite are in most collectors' cabinets.

An ardent mineralogist, the late Dr. Foote, in cutting a section of this meteorite, found the tools were injured by something vastly harder than metallic iron, and an emery wheel used in grinding the iron had been ruined. He examined the specimen chemically, and soon after announced to the scientific world that the Canyon Diablo meteorite contained black and transparent diamonds. This startling discovery was afterwards verified by Professors Friedel and Moissan, who found that the Canyon Diablo meteorite contained the three varieties of carbon—diamond (transparent and black), graphite, and amorphous carbon. Since this revelation, the search for diamonds in meteorites has occupied the attention of chemists all over the world.

I am enabled to show you photographs of true diamonds I myself have extracted from pieces of the Canyon Diablo meteorite, five pounds of which I have dissolved in acids for this purpose—an act of vandalism in the cause of science for which I hope mineralogists will forgive me. A very fine slab of the meteorite, weighing about seven pounds, which has escaped the solvent, is on the table before you.

Here, then, we have absolute proof of the truth of the meteoric theory. Under atmospheric influences the iron would rapidly oxidise and rust away, colouring the adjacent soil with red oxide of iron. The meteoric diamonds would be unaffected, and would be left on the surface of the soil to be found by explorers when oxidation had removed the last proof of their celestial origin. That there are still lumps of iron left at Arizona is merely due to the extreme dryness of the climate and the comparatively short time that the iron has been on our planet. We are here witnesses to the course of an event which may have happened in geologic times anywhere on the earth's surface.

Although in Arizona diamonds have fallen from above, confounding all our usual notions, this descent of precious stones seems what is called a freak of Nature rather than a normal occurrence. To the modern student of science

there is no great difference between the composition of our earth and that of extra-terrestrial masses. The mineral peridot is a constant extra-terrestrial visitor, present in most meteorites. And yet no one doubts that peridot is also a true constituent of rocks formed on this earth. The spectroscopic reveals that the elementary composition of the stars and the earth are pretty much the same; so does the examination of meteorites. Indeed, not only are the selfsame elements present in meteorites, but they are combined in the same way to form the same minerals as in the crust of the earth.

This identity between terrestrial and extra-terrestrial rocks recalls the masses of nickeliferous iron of Ovifak. Accompanied with graphite they form part of the colossal eruptions which have covered a portion of Greenland. They are so like meteorites that at first they were considered to be meteorites till their terrestrial origin was proved. They contain as much as 1·1 per cent of free carbon.

It is certain from observations I made at Kimberley, corroborated by the experience gained in the laboratory, that iron at a high temperature and under great pressure will act as the long-sought solvent for carbon, and will allow it to crystallise out in the form of diamond—conditions existent at great depths below the surface of the earth. But it is also certain, from the evidence afforded by the Arizona and other meteorites, that similar conditions have likewise existed among bodies in space, and that a meteorite freighted with its rich contents, on more than one occasion has fallen as a star from the sky. In short, in a physical sense, Heaven is but another name for Earth, or Earth for Heaven.

A NEW FORM OF REFLECTING TELESCOPE.

By CHARLES LANE POOR.

Dr. Poor explained some experiments that he has carried out in grinding and polishing a new form of parabolic mirror for reflecting telescopes. The mirror is a portion of a paraboloid of revolution cut at the extremity of the latus rectum. The reflected beam is at right-angles to the incident light; no second mirror is therefore necessary; the full aperture of the mirror being used.

The advantages of such a form of mirror were pointed out, and the great simplification in equatorial mountings indicated. The declination axis becomes the telescope tube, the image being formed at the intersection of the polar and declination axis and is always in the same position; the observer remains at rest while viewing any and every part of the visible heavens. No dome is required. Other advantages were indicated, and several modifications of the general form pointed out—*Johns Hopkins University Circular*, xvi., No. 130.

TESTIMONIAL TO PROF. ATTFIELD, F.R.S.

An interesting ceremony was performed on Saturday afternoon last, the 10th July, when Prof. Attfield was presented with a testimonial on the occasion of his retirement from the Chair of Practical Chemistry in the School of the Pharmaceutical Society of Great Britain, after having occupied the position for thirty-four years.

The testimonial took the form of an album containing the signatures of one thousand old pupils and two hundred other friends of Prof. Attfield, a silver tray, and a silver tea and coffee service—all of which were suitably inscribed.

The presentation was made at Prof. Attfield's residence, at Ashlands, Watford, where Mrs. Attfield had a garden-party and reception. We trust he will live long to enjoy the rest he has so well earned.

HYPOIDOUS ACID AND HYPOIODITES.*

By R. L. TAYLOR, F.C.S.

(Concluded from p. 20).

Hypiodous Acid.

So far as I have been able to ascertain, it has always been stated that all attempts to obtain hypiodous acid by the action of iodine and water upon mercuric oxide have failed, and that nothing but iodic acid was formed.† I had tried the action once more with the aqueous solution of iodine, and had apparently been as unsuccessful as ever. I had succeeded, as I believe, in obtaining the acid by other methods, to be presently described, and had noticed the curious anomaly that the free acid, or what I took to be the free acid, bleached indigo with far less energy than the hypiodites described in the first part of this paper; but I had failed, as others no doubt had frequently failed, to obtain any bleaching solution by the action of iodine and water upon mercuric oxide.

In the paper already referred to by Walker and Kay, the authors state that they "made a solution of hypiodous acid by agitating pure aqueous solution of iodine with mercuric oxide," filtered, neutralised with potash, and added magnesium sulphate, so obtaining a white precipitate. The addition now of a few drops of potassium iodide stained the precipitate brown. This certainly pointed to the presence of a hypiodite, which would be decomposed, with liberation of iodine, by potassium iodide. I thought the reaction might possibly be due to iodic acid, which is usually said to be the sole product of the action of iodine on mercuric oxide; but experiment convinced me that it was not. Further investigation soon showed that hypiodous acid is really produced when iodine water is shaken up with mercuric oxide (the precipitated oxide is far the best) and filtered. Many others besides Walker and Kay have no doubt prepared the acid in this way, but failed to recognise it. The explanation is that the bleaching action of the free acid is *excessively feeble* as compared with Schönbein's solutions. Contact with the indigo solution for a long time causes the colour slowly to disappear, but the addition of a drop of alkali immediately transforms the acid into as strong a bleaching solution as Schönbein's solutions.

I may at once state what appears to me a possible explanation of what seems at first sight a most extraordinary anomaly—that a free acid has a very much feebleness oxidising power than one of its salts! When hypiodous acid bleaches, I suppose it does so according to the following equation:— $\text{HOI} = \text{HI} + \text{O}$. It thus produces hydriodic acid, an extremely unstable body itself, and, further, a compound which would immediately decompose the remaining hypiodous acid. On the other hand, in presence of an alkali, the result of losing oxygen would be to produce sodium or potassium iodide, both perfectly stable bodies, and with any tendency to decom-

* From *Memoirs and Proceedings of the Manchester Literary and Philosophical Society*, vol. xli., Part III.

† So long ago as 1845, Köne (*Poggendorff's Ann.*, lxvi., p. 302) tried the experiment of shaking up precipitated mercuric oxide with an alcoholic solution of iodine, and, from the fact that he obtained an unstable solution which gradually liberated iodine, and from analogy with the chlorine compounds, he concluded that hypiodous acid was formed. I have repeated the experiment, and there certainly appears to be a hypiodous compound produced; the alcohol which is present, however, interferes with the reactions. The filtered liquid contains a considerable amount of mercury. It gives a yellowish precipitate with water, and if a little alkali is added to this it possesses very strong bleaching properties. The precipitate produced with water dissolves up in sodium hydrate, but in a few seconds another precipitate appears which is manifestly iodoform; at the same time the liquid loses its bleaching power. This appears to point to the conclusion that the formation of a hypiodide is the necessary prelude to the formation of iodoform (see Van Deventer and van't Hoff, *Chem. Central.*, 1888, p. 362). On account of this rapid change in presence of an alkali, the solution does not give the cobalt reaction. If the precipitate produced by water in the alcoholic solution is allowed to stand for some hours, scarlet mercuric iodide separates out. The alcoholic solution is moderately stable, but it gradually liberates iodine on standing.

pose the remaining hypoiodite counteracted by the presence of the alkali. Then there is also the further consideration that apparently hypoiodous acid can only have about one-half the total bleaching power that a hypoiodite will have, because, as soon as any of it does bleach it produces hydriodic acid, which would immediately decompose an equivalent amount of the remaining hypoiodous acid. (In one comparative experiment that I made, the bleaching power of the free acid was almost exactly half that of an equal volume of the same iodine solution to which soda had been added. But the bleaching continued for *two hours*, so that the effect would be complicated by the spontaneous decomposition of the free acid).

When a little alkali is added to the hypoiodous acid prepared in this way, the solution behaves almost exactly like Schönbein's solutions. It bleaches strongly, and some determinations I have made with the standard indigo solution gave a bleaching action equivalent to 80 per cent of the iodine used,—that is, representing 40 out of a possible 50 per cent of iodine existing as hypoiodous acid. This result again is confirmed by experiments by Schwicker's method, only in the case of this solution, when it is neutralised by an alkali, it forms nothing else but hypoiodite, and consequently the addition of soda-water simply liberates hypoiodous acid, and there is no separation of iodine. In order to complete the determination potassium iodide has to be added, when there is an immediate liberation of iodine. A determination by this method, which is probably more accurate than the bleaching method, gave liberated iodine equal to 90 per cent of that originally used, so that 45 out of a possible 50 per cent of iodine existed in the solution as hypoiodous acid.

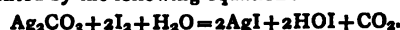
The hypoiodous acid solution to which a little alkali has been added gives a precipitate with cobalt solution, which gradually turns brown and then black, and an immediate brown precipitate with a manganous salt. With silver nitrate it gives a buff-colored precipitate similar to the one I had previously obtained from Schönbein's solutions, but in this case of course it will not be mixed with so much silver iodide. If nitrate of silver is added to the solution containing the free acid a milkiness is produced, and on boiling there is a further precipitate, which contains silver iodide and iodate; the iodate can be dissolved out by ammonia. This reaction I had previously noticed with the acid prepared in another way. The hypoiodous acid is manifestly converted into hydriodic and iodic acids, $3\text{HIO} = 2\text{HI} + \text{HIO}_3$. The aqueous solution of the free acid appears to be moderately stable. When it decomposes, as one would anticipate, it appears to do so according to the equation given above, only in this case the two acids immediately decompose each other, liberating iodine.* In the case of the solution made, as described, with the use of aqueous solution of iodine, the appearance of free iodine in the liquid is very slow; but, by using iodine-water with a little suspended precipitated iodine, a much stronger solution of the hypoiodous acid is obtained, in which the brown colour of iodine begins to show itself within a minute or two of its being filtered. (It appears also that some mercury comes through, because on standing for some hours there is a slight deposit of the scarlet iodide of mercury). After standing for some time, this solution, which manifestly contains free iodine (carbon bisulphide shaken up with it is coloured deep violet), gives no blue colour with starch until the addition of a drop of alkali, or until it has been exposed to the air for some time. If a little of the mixture of the brown solution with starch is poured into a shallow

vessel, or is poured backwards and forwards from one vessel to another, it soon turns blue.*

Some time before obtaining the free hypoiodous acid in the way described above, I had obtained what must have been either the acid or a solution of its silver salt by another method. It was pointed out by Dancer (*Chem. Soc. Journ.*, xv., 447) that hypobromous acid could be obtained by the action of bromine-water upon solution of nitrate of silver, according to the following equation:—



Chlorine acts in a similar way, producing hypochlorous acid. A solution of nitrate of silver with powdered iodine appears to form nothing but iodide and iodate of silver; but I found that iodine suspended in water gave a bleaching liquid when shaken up with solution of silver sulphate, or with a paste of silver carbonate, the carbonate giving the better result. Wishing to obtain some quantitative results, I began to use an aqueous solution of iodine, instead of having it merely suspended in water. Shaking up this solution with a little silver carbonate and rapidly filtering, a liquid is obtained which contains a small amount of silver, but which gives all the reactions which I have described as characteristic of Schönbein's solutions, complicated a little, in some cases, by the silver which is present. It bleaches indigo slowly, but much more rapidly than the acid prepared with mercuric oxide; it also bleaches cochineal and logwood, and oxidises cobalt and manganese salts in presence of an alkali. The action of the iodine upon the silver carbonate may probably be represented by the following equation:—



If a drop of silver nitrate is added to the solution and the liquid boiled, a yellow precipitate is produced, containing iodide and iodate of silver.

I made many attempts to ascertain, by various methods, the proportion of the iodine used which was converted into hypoiodous acid. I filtered the liquid into an acidified solution of potassium iodide, whereby iodine is liberated, the amount of which I estimated. Another method tried was to allow the liquid to run into a standard solution of sulphurous acid, and then to find the amount of this which was oxidised. The results were not satisfactory, as I seldom found that more than 50 per cent of the iodine was converted into bleaching iodine. I have reason to believe that this result was due to some insoluble hypoiodite of silver being left in the precipitate. Finally I tried the standard indigo solution, and found that it was possible to estimate the bleaching power even in the muddy liquid containing the excess of silver carbonate and the silver iodide which was produced in the reaction. Shaking up a known volume of the iodine solution with silver carbonate and immediately running in the indigo solution, the bleaching action indicated about 50 to 60 per cent of the theoretical amount; but the addition of a few drops of dilute sulphuric acid carried the bleaching power still further, owing no doubt to the decomposition of some insoluble silver hypoiodite by the acid. By adding the dilute acid immediately after shaking together the iodine and the silver carbonate, a bleaching action was obtained equal to from 90 to 95 per cent of the theoretical amount. Subsequently I found that, with the aqueous solution of iodine, nitrate of silver reacts perfectly well, producing a bleaching liquid which gives all the reactions already described, complicated a little by the presence of excess of silver. The bleaching action, immediately after the silver nitrate has been added, indicates 95 per cent of the theoretical amount.

The solutions prepared by the use of these silver salts

* It will be somewhat difficult to measure the rate at which the hypoiodous acid decomposes. Neither of the two methods mentioned in the text for estimating the amount of hypoiodite is applicable when once the decomposition of the acid has started, because each of them would require the preliminary addition of a little alkali, and this would at once convert any liberated iodine into iodide and hypoiodite.

* Dr. A. Harden tells me that he has found that a mixture of aqueous iodine and iodic acid, in certain proportions, behaves in exactly the same way as the above solution. It is known that iodine forms no blue compound with starch unless an iodide be present; but I can offer no explanation of the effect which the air appears to have on the above reaction.

are extremely unstable; that made with the silver nitrate loses 90 per cent of its bleaching power on standing five minutes. This I think may be attributed to the presence of silver in the solution, and to the tendency of the iodine and silver to form the insoluble silver iodide. The solutions prepared in this way are considerably more sluggish in their bleaching action than the alkaline hypiodites, but very much more rapid than the free acid prepared by the mercuric oxide method. If the explanation which I suggested for the difference between the free acid and Schönbein's solutions is correct, it seems to me that it would apply in this case as well. There is silver present in the solutions, and therefore when the bleaching is finished the final product will be silver iodide, a much more stable body than hydriodic acid.

I have to thank Mr. G. P. Varley, B.Sc., and Mr. J. H. Wolfenden, B.Sc., for assistance given me in some portions of this work.

ON THE VOLUMETRIC DETERMINATION OF ZINC BY POTASSIUM FERROCYANIDE.

By L. L. DE KONINCK and EUG. PROST.

(Continued from p. 25).

E.—THE zincic solution is placed in a matrass graduated to 200 c.c.; we add 25 c.c. of ferrocyanide, and fill with water up to the mark, shake well, then let stand. The precipitate behaves as in experiments A and B. After standing three hours, we take 100 c.c. of the clear liquid and titrate directly, in the cold, with ferrocyanide solution, until the mixture when tested with uranium gives a very faint brown tint, even if the test is repeated after a few minutes. Using 11.30 c.c. with an extra two drops of ferrocyanide, the uranium test gives a distinct colouration, showing an excess of the reagent. Total ferrocyanide used for 25 c.c. of zincic solution:—

$$25 + (2 \times 11.30) = 47.60,$$

instead of 50 c.c., the theoretical quantity. The importance of this test, from the point of view of obtaining an exact knowledge of the reaction, decided us to repeat it under slightly different conditions.

E'. We used the same zincic solution, $\frac{1}{2}$ normal, but a solution of ferrocyanide, $\frac{1}{2}$ normal for zinc,—that is to say, corresponding exactly to that of zincic chloride under the conditions of the formation of $K_2Zn_3Fe_2Cy_{12}$: 25 c.c. of the zincic chloride, diluted with 100 c.c. of water acidulated with hydrochloric acid, are placed in a flask graduated to 200 c.c.; we run in 13 c.c. of ferrocyanide, and fill with water up to the mark. The precipitate is very gelatinous, and remains so. After standing forty-eight hours it has not changed, and still occupies two-thirds of the total volume; the supernatant liquid is perfectly clear.

Filter off 100 c.c. of clear liquid (I.). That part of the precipitate remaining on the filter is completely removed; the filter is washed, and the washings added to the part remaining in the flask (II.) This experiment was done in duplicate.

I. To determine the quantity of zinc remaining in (I.) we added 6 c.c. of ferrocyanide. After digesting for fifteen minutes the precipitate becomes of an opaque white colour; we found with nitrate of uranium the presence of an excess of the reagent, which is titrated back with chloride of zinc.

	First test.	Second test.
$ZnCl_2$ — $\frac{1}{2}$ normal used ..	1.70 c.c.	1.75 c.c.

The end of the reaction is shown very sharply.

II. To the troubled liquor (II.) we first add, as a preliminary, 6 c.c. of ferrocyanide; an immediate uranium test gives an intense reaction, but after a few instants no

reaction at all. Add a further 2 c.c. of ferrocyanide; an immediate test again gives a very marked reaction, but after a few seconds only a faint tint, until, after a further addition of 1 c.c., the test shows an excess of the reagent even after standing for some minutes.

In the second parallel operation we used at once 9 c.c. of ferrocyanide. On the precipitate becoming white, we titrated back with zincic chloride after a lapse of twenty minutes.

	First test.	Second test.
$ZnCl_2$ — $\frac{1}{2}$ normal used ..	1.25 c.c.	1.35 c.c.

The end of the reactions was not so distinctly marked; this we attribute to the relatively large quantity of the precipitate.

The conclusion to be drawn from the experiments E and E' is that, even in the presence of a large excess of zincic salt (50 and 48 per cent), the precipitate produced by the ferrocyanide is formed principally of zincic-potassic ferrocyanide, but contains nevertheless zincic ferrocyanide; this latter reacts with the potassic salt, and combines with it so as to form $K_2Zn_3Fe_2Cy_{12}$.

If the ferrocyanide is in sufficient proportion, or, better still, in excess, the precipitate is formed exclusively of the compound $K_2Zn_3Fe_2Cy_{12}$. At the moment of its formation this body is gelatinous, and reacts sharply with salts of uranium; but it aggregates rapidly, and soon gives no further such reaction.

Formed in presence of an excess of zinc, the precipitate contains a small proportion of zincic ferrocyanide; that is, at least, what appears to result from the last experiment, since titration only detected respectively 90.4, 71.7, and 70.8 per cent of the quantities which should be present if the precipitate had been formed solely of $K_2Zn_3Fe_2Cy_{12}$.

But whatever may be the form under which this excess of zinc exists in the first precipitate, it still reacts with the ferrocyanide in such a manner as to finally produce $K_2Zn_3Fe_2Cy_{12}$, but without doubt more slowly than if it were in solution.

IV. Research on the best Method of Working to be used.

It would appear, from what has preceded, that the reaction would be more regular, and its termination more sharply marked, if we proceeded in such a manner that the ferrocyanide should be always, as far as possible, in excess with regard to the zinc,—that is to say, if we added the zincic solution to the ferrocyanide, instead of running this latter into that containing the zinc; this has already been noted by Zulkowski. To effect this it is necessary, in estimating zinc, either to operate, by inverse titration, which is not very practicable, or else to titrate back. This method of working has, further, an advantage observed by Mohr (*Mohr-Classen*, 1886, p. 458) of letting us know when we are approaching the end of the reaction, this being shown by the diminution of intensity of the colouration produced by the indicator, while in direct titration the termination is shown suddenly, and it is easy to overshoot the mark.

For these reasons we have given the preference to titrating back.

We would wish, however, to mention what accuracy can be obtained by direct titration. Our experiments in this direction having soon brought us to the conclusion that titrating back is much more preferable, we will not go into much detail, but will content ourselves with saying that working in the cold, either in hydrochloric or acetic solution, the end of the reaction is very indistinct; thus, as we have already shown, the drop test, done immediately after the addition of the ferrocyanide, gives a distinct colouration, diminishing in clearness as we leave a longer time between the addition of the liquid and taking the drop. By stopping the estimation at the moment when the nitrate of uranium begins to show, we, as a rule, obtain results which cannot be called very exact, varying considerably with the conditions of the experi-

ment,—that is to say, with the state of the precipitate; if the operation is carried out warm, the reaction is sharper. By working slowly, under definite conditions, especially using heat, we can, however, obtain results which might be considered satisfactory if we did not possess, in "titrating back," a much more satisfactory method.

Titration back with permanganate of potash has already been proposed by Renard (*Comptes Rendus*, vol. lxxvii., p. 450, 1868), but this method necessitates the removal of the precipitate by filtration, and the zincic or zincicopotassic ferrocyanide reacts with the permanganate. The back titration by means of a solution of zincic chloride enabled us to obtain very satisfactory results, either with solutions of salts of pure zinc or with minerals. It now remains to establish by experiment the most favourable conditions of working.

Influence of Time.—From what we have seen, a great deal depends on whether we perform the "touch" test with nitrate of uranium, immediately after the mixture of the zincic salt and the ferrocyanide, or whether we wait for a few minutes. It was therefore necessary to determine the minimum time, after which the test gives constant results.

In the following experiments we proceeded each time by pouring 20 c.c. ZnCl_2 , $\frac{1}{2}$ normal, into a mixture containing—

50 c.c. ferrocyanide, $\frac{1}{2}$ normal for zinc;
10 c.c. HCl , 5 normal;
50 c.c. AmCl , at 20 per cent; *
100 c.c. water.

Back titration by the same $\frac{1}{2}$ normal solution of zincic chloride:—

1. Titration done as rapidly as possible, used for this 5.50 ZnCl_2 ; total, 25.50.
2. Titration done at once, but without undue haste, used 5.00 ZnCl_2 ; total 25.00.
3. Titration after 7.5 minutes, used 4.65 ZnCl_2 ; total, 24.65.
4. Titration after 15 minutes, used 4.55 ZnCl_2 ; total, 24.55.
5. Titration after 30 minutes, used 4.55 ZnCl_2 ; total, 24.55.

Fifteen minutes therefore suffice to obtain a complete transformation of the precipitate.

Influence of the Initial Excess of Ferrocyanide.—We may ask if a notable excess of potassic ferrocyanide is necessary to transform, integrally and rapidly, into a double cyanide the small quantity of zincic ferrocyanide which seems to form when we mix the alkaline ferrocyanide and the zinc solution. The following series of experiments answer this question:—Five solutions were prepared, each containing, as in the preceding series, 10 c.c. HCl , 5 normal; 50 c.c. AmCl at 20 per cent, and 100 c.c. of water; then respectively 5, 10, 15, 20, and 24 c.c. of ZnCl_2 , $\frac{1}{2}$ normal. Into each was run 50 c.c. of $\frac{1}{2}$ normal ferrocyanide, and titrated back after a quarter of an hour's digestion. This required respectively—

19.50, 14.55, 9.50, 4.45, 0.80 ZnCl_2 ,

or a total, for the 50 c.c. of ferrocyanide, of—

24.50, 24.55, 24.50, 24.45, 24.80 ZnCl_2 .

We see from these results that an excess corresponding to 5 c.c. on 25, or, 20 per cent is quite sufficient to produce the transformation in fifteen minutes. The only result which is notably away from the average is that where the excess of ferrocyanide corresponds to less than 1 c. of the zincic solution.

Influence of the Order of Mixing.—It is indifferent whether the zinc solution is poured into the ferrocyanide

or *vice versa*. We have in many assays, worked either one way or the other, without noticing the slightest difference in the results, or even in the rapidity with which the molecular transformation of the precipitate takes place. Special tests have moreover proved it.

Influence of Chloride of Ammonium.—In each of the experiments in the two following series we used 20 c.c. ZnCl_2 , $\frac{1}{2}$ normal; 50 c.c. of ferrocyanide, $\frac{1}{2}$ normal; 10 c.c. of hydrochloric acid, 5 normal; and 150 c.c. of a mixture of water and a 20 per cent solution of ammonium chloride, in the proportions given below, titrating back with the same zincic solution.

AmCl.	H_2O .	ZnCl_2 .		ZnCl_2 Total.	
		Series I.	Series II.	Series I.	Ser. II.
1. 0	150	5.05	—	25.05	—
2. —	—	5.00	4.80	25.00	24.80
3. 25	125	4.75	4.40	24.75	24.40
4. 50	100	4.55	4.33	24.55	24.33
5. 75	75	4.60	4.24	24.60	24.24
6. 100	50	4.50	4.10	24.50	24.10
7. 125	25	4.55	4.05	24.55	25.05
8. 150	0	4.75	3.85	24.75	23.85

(To be continued).

PRECIPITATION OF COPPER BY MAGNESIUM.

By E. G. BRYANT.

I was induced, by a paragraph in the South Kensington Chemistry Syllabus, to try some experiments on the action of magnesium on solutions of copper salts, and though the method used was necessarily extremely crude, the results seemed so decisive that I thought they might be of interest to others. It was intended that the work, if successful, should be attempted by mere beginners in practical chemistry; therefore the method used was made as simple as possible, all filtering being dispensed with. The materials used were commercial magnesium ribbon, copper sulphate, and chloride. The magnesium was thoroughly cleaned and the CuSO_4 re-crystallised. After precipitation, the copper was well washed by decantation, dried, and weighed either as copper or copper oxide. My experience seems to show that it is easier, unless a furnace or foot blowpipe be at hand, to get good results with metallic copper than with the oxide, as an ordinary Bunsen takes a considerable time to oxidise even a hundred or so milligrams of the metal.

To show the degree of accuracy which may be obtained under such circumstances, 107.7 m.grms. of Cu were obtained by placing 105.2 m.grms. of zinc in a warm solution of copper sulphate, which gives an equivalent of 62.8 for the copper.

I have made a large number of experiments with magnesium and copper sulphate and chloride, using both dilute and concentrated solutions, at about 18°, 50°, and 90° C., also solutions made alkaline with ammonia and sodium hydrate, but invariably with unsatisfactory results. The copper or copper oxide obtained varies greatly in amount, but is never much above 70 per cent of the theoretical quantity. The highest results were given by hot concentrated solutions; in dilute or cold solution the process required hours to complete, and the results were under 50 per cent. In every case a considerable amount of hydrogen was generated, and more or less of the magnesium became oxidised. The magnesium oxide in turn displaced copper hydrate, so that a greenish sediment was always found accompanying the precipitated copper. This result by itself would be enough to prove that the reaction was not a quantitative one. Very dilute hydrochloric acid was used to dissolve this greenish substance, and a small quantity of the copper was removed at the same time (not more than 8 to 10 per cent).

* We showed by later experiments, described further on, the influence of the presence of chloride of ammonium; we added it in a large number of experiments, so as to get as near as possible to the conditions of mineral analysis.

I give one or two of my results:—

Mg.	Cu.	Cu (theoretical).	
0.055 grm.	0.079	0.144	Strong solution at about 50° C. One hour required.
0.055 "	0.064	0.144	Strong solution at 18° C. Several hours.
0.065 "	0.137	0.171	Conc. sol. at about 80° C. A few minutes only.

The results obtained from perfectly pure magnesium might be very different from the above, but I have not been able to use any but the commercial metal.

An excess of magnesium oxide removes every trace of copper from solutions of the sulphate and chloride, and a bluish residue is obtained containing copper hydrate (?) and magnesium oxide. It settles readily, filters easily, and is unchanged by long boiling, or by exposure to air or water for some weeks.

ANALYSIS OF BRONZES AND BRASSES BY THE ELECTROLYTIC PROCESS.

By A. HOLLAND.

THE object of this communication is to give in detail the procedures for the accurate and easy determination of copper, tin, zinc, &c., entering into the composition of bronzes and brasses.

I. Bronzes.

Determination of Copper (Electrolysis in Acid Solution).

—Five grms. of alloy are treated in a beaker (Bohemian glass) with a mixture of 25 c.c. nitric acid, at sp. gr. 1.31°, and 15 c.c. concentrated sulphuric acid. In presence of so large a proportion of sulphuric acid the tin dissolves, at least in part. We dilute to 350°, and heat the liquid to a temperature close upon ebullition, keeping it at this temperature until the insoluble part which contains the tin has collected at the bottom of the vessel. We thus obtain a perfectly clear liquid, into which we can plunge without causing turbidity, the platinum cone and spiral serving as electrodes. For the progress of the electrolysis we follow the indications given in *Comptes Rendus*, cxixiii, p. 1003.

Determination of Tin (Electrolysis in Hydrochloric Solution, with the addition of Ammonium Oxalate).

—The liquid, free from copper, is evaporated on the sand-bath until there remain only a few drops of sulphuric acid. The residue is taken up in hydrochloric acid and water, and the tin is precipitated by a current of sulphuretted hydrogen in the ordinary manner. The tin sulphide, washed as usual with a solution of sodium chloride, is dissolved in yellow ammonium hydrosulphate, and this solution is evaporated to dryness on the water-bath. The residue obtained is attacked with 9 grms. of potassium chlorate dissolved in water and an excess of hydrochloric acid. The solution of tin thus obtained is again evaporated to dryness on the water-bath, and the residue taken up in 30 c.c. of hydrochloric acid and water. We filter this new solution, and dissolve in it 30 grms. of pure ammonium oxalate; and lastly, heated to about 90° and electrolysed, using a current of 0.7 ampère. At the end of twelve hours the deposition is complete, and the deposit is strongly adhesive.

Determination of Zinc by Electrolysis.—The liquid, free from copper and tin, is heated to expel all the hydrogen sulphide in solution, then evaporated to dryness on the sand-bath until there remain only a few drops of sulphuric acid. The zinc sulphate thus formed is dissolved in water, neutralised with ammonia, and to the solution there are added 15 grms. of ammonium citrate and 4 c.c.

glacial acetic acid, ammonia up to neutralisation, and, lastly, 3 c.c. crystallisable acetic acid.

The bath thus obtained contains, besides zinc in the state of sulphate, ammonium, acetate and citrate, and acetic acid. It is exposed for about twelve hours to a current of 0.6 ampère. At the end of this time all zinc is deposited upon the cone as a very adhesive deposit.

If the bronze contains iron, this will be deposited with the zinc, in part at least. In this case the weight of the iron (determined by permanganate) is deducted from the weight of the zinc thus found.

Lead, which is often met with in bronzes, is determined by electrolysis in a fresh nitric solution.

II. Brasses.

Copper is determined as in *Comptes Rendus*, cxixiii, p. 1003. The determination of zinc and of impurities is effected by the procedures given above.—*Comptes Rendus*, cxixiv, p. 1451.

ON CERTAIN DOUBLE HALOGEN SALTS OF CÆSIUM AND RUBIDIUM.

By H. L. WELLS and H. W. FOOTE.

1. The Complicated Rubidium-antimony Chloride.

REMSEN and Saunders (*Am. Chem. Journ.*, xiv., 155) have described a salt to which they gave the formula $23\text{RbCl} \cdot 10\text{SbCl}_3$, as the most probable one. Wheeler (*Am. Journ. Sci.*, xlv., 269), working in the Sheffield Chemical Laboratory, New Haven, confirmed Remsen and Saunders's results and discovered besides an analogous bromide, to which the probable formula $23\text{RbBr} \cdot 10\text{SbBr}_3$ was given. Remsen and Brigham (*Am. Chem. Journ.*, xiv., 174) prepared the salt $23\text{RbCl} \cdot 10\text{BiCl}_3$. Herty (*Am. Chem. Journ.*, xvi., 490) has since described the two potassium salts, $23\text{KCl} \cdot 10\text{SbCl}_3$ and $23\text{KBr} \cdot 10\text{SbBr}_3 \cdot 27\text{H}_2\text{O}$, and some mixtures of these two salts.

In view of all this work there can scarcely be a doubt as to the existence of a type of salts with a somewhat complicated ratio, but in view of the fact that this complicated ratio 23:10 is apparently an exception to the simplicity of composition of all other carefully investigated double halogen salts, the subject seemed worthy of some further investigation. For the purpose, we have studied only the rubidium-antimony chloride of Remsen and Saunders, as this salt is readily prepared and is capable of repeated re-crystallisation from hydrochloric acid solution.

The possibility suggested itself that the product might consist of two simpler salts of similar or identical crystalline form, which were capable of crystallising together, and that previous investigators had made use of conditions which resulted in obtaining a constant mixture of two such salts. Although this supposition had scarcely any probability in view of the existence also of the rubidium-antimony bromide and of the two potassium salts, we have put the question to test by repeatedly re-crystallising the salt, using not only ordinary dilute hydrochloric acid for this purpose, but also more dilute and much more concentrated acid and also an alcoholic hydrochloric acid solution. As will be seen from the analyses, given beyond, no variation in composition could be detected by the use of these widely varying solvents for re-crystallisation, and it therefore appears impossible that the salt can be a mixture.

As a starting-point, we used a solution in hydrochloric acid containing the constituents RbCl and SbCl_3 in the exact molecular proportion 23:10. Product A was the first, B the third, and C the fifth re-crystallisation from pure dilute hydrochloric acid. The product D was obtained by adding concentrated hydrochloric acid to a

nearly saturated warm solution of the salt in dilute hydrochloric acid. E was obtained from a very strong hydrochloric acid solution formed by passing a rapid current of hydrogen chloride gas into the solution as it cooled. F was obtained by re-crystallising the salt from hydrochloric acid which was kept as dilute as it could be without producing the basic double salt to be described beyond. G was a product obtained by re-crystallising the salt from a mixture of equal volumes of dilute hydrochloric acid and alcohol.

The two products obtained from concentrated hydrochloric acid solution had a pale yellow colour, while the others were all white. The crystals were usually well-formed six-sided plates which showed no definite optical properties.

The analyses of the various products are as follows:—

	Rubidium.	Antimony.	Chlorine.
A	39'23	23'85	37'01
B	39'23	23'84	36'99
C	—	23'91	—
D	39'25	23'98	—
E	39'31	23'89	—
F	39'03	23'86	—
G	39'11	23'90	—
Average	39'19	23'89	37'00

Method of Analysis.—For the determination of antimony and rubidium, a portion of about $\frac{1}{4}$ gm. was dissolved in water and enough hydrochloric acid to prevent antimony oxychloride from precipitating. The solution was heated to boiling and hydrogen sulphide passed in. The solution was then cooled, and the antimony sulphide filtered on a Gooch crucible and washed with water and with alcohol. The crucible was then slowly heated to 230° and cooled in an oven filled with carbonic acid. The precipitate was weighed as Sb_2S_3 . The filtrate containing rubidium was evaporated with sulphuric acid and the residue ignited in a stream of air containing ammonia, and weighed as Rb_2SO_4 . Chlorine was determined by dissolving a separate portion in water acidified with tartaric and nitric acids and precipitating with silver nitrate. This was allowed to stand for some time and the precipitate was then collected on a Gooch crucible and weighed. The methods used are almost identical with those of Wheeler.

The accuracy of the antimony determination was checked in the following manner:—The salt, $Cs_3Sb_2Cl_9$, was prepared from very pure materials and carefully re-crystallised, and antimony determined by the above method. The per cent of antimony is nearly the same as in the rubidium-antimony salt under consideration. The following results were obtained:—

	I.	II.	III.	IV.
Per cent Sb found	25'37	25'42	25'43	25'44
„ „ calculated	25'13	—	—	—

The atomic weights used in all the calculations were Rb, 85'43; Sb, 120'43; Cl, 35'45; S, 32'07; Ag, 107'92; Cs, 132'89.

Since the method used for the determination of antimony gives results which are slightly too high, we believe that a deduction of the average error 0'25 per cent from the antimony found in the analyses of the rubidium salt will give a result which is nearer the truth.

	Rb.	Sb.	Cl.
Average previously given	39'19	23'89	37'00
Average with correction for Sb	39'19	23'64	37'00
Calculated for $Rb_{23}Sb_{10}Cl_{53}$	38'92	23'86	37'22
Calculated for $Rb_7Sb_3Cl_{16}$	39'18	23'66	37'16

It may be noticed that the results agree rather more satisfactorily with the formula $7RbCl_3SbCl_3$ than with the more complicated one advanced by Remsen and Saunders. The differences between these formulæ are,

however, so slight that it is probably entirely impossible to decide between them by means of chemical analysis, the ratio Rb: Sb being 230: 100 in one case, and in the other 233: 100. However, since it is customary to use the simplest applicable formula for a chemical compound, we propose the formula $7RbCl_3SbCl_3$ for this salt, and corresponding formulæ for other salts of this series. Herty's hydrous salt, to which he gave the formula $23KBr \cdot 10SbBr_3 \cdot 27H_2O$, agrees well with the formula $7KBr \cdot 3SbBr_3 \cdot 8H_2O$. It must be admitted that the 7: 3 ratio is an unusually complicated one, but it is far simpler than 23: 10, and is scarcely a marked exception to the general simplicity of double halogen salts.

2. A Rubidium-antimony Oxychloride, $2RbCl \cdot SbCl_3 \cdot SbOCl$.

In attempting to re-crystallise the salt $7RbCl_3SbCl_3$ from very dilute hydrochloric acid, just enough to prevent the formation of antimony oxychloride, this new salt was obtained in the form of short colourless prisms possessing a rather high lustre. It can be re-crystallised from very dilute hydrochloric acid.

The following results were obtained from analyses of separate crops:—

	I.	II.	Calculated for $2RbCl \cdot SbCl_3 \cdot SbOCl$.
Rb	26'54	26'68	26'68
Sb	37'58	37'36	37'61
Cl	32'75	32'80	33'21
O (by diff.)	3'13	3'16	2'50

It is interesting to notice that Benedikt (*Proc. Am. Acad.*, xxix., 212) has described the potassium salt $2KCl \cdot SbCl_3 \cdot SbOCl$, which corresponds exactly to this rubidium compound.

3. The Cæsium-bismuth Chlorides and Iodides.

The double chlorides of bismuth with cæsium have been described by Remsen and Brigham (*Am. Chem. Journ.*, xiv., 179). These authors did not state, however, how widely the conditions had been varied, and we have repeated the work, varying the proportions of cæsium and bismuth as much as possible, and have found exactly the same salts as described by them. These salts are,—



$3CsCl \cdot BiCl_3$.—This salt forms in colourless plates when 50 grms. of cæsium chloride are mixed in hydrochloric acid solution with from 1 to 25 grms. of bismuth chloride. The analyses were made on samples, dried but a short time in the air, which apparently contained a little mechanically included water. The following results were obtained:—

	I.	II.	Calculated for $3CsCl \cdot BiCl_3$.
Bi	24'80	24'47	25'36
Cs	47'94	—	48'66
Cl	—	—	25'98

$3CsCl \cdot 2BiCl_3$.—When 50 grms. of bismuth chloride are mixed with from 1 to 80 grms. of cæsium chloride, the salt $3CsCl \cdot 2BiCl_3$ crystallises in light yellow needles, sometimes broadening and looking like plates and again much shorter and thicker.

The following analyses were made:—

	I.	II.	Calculated for $3CsCl \cdot 2BiCl_3$.
Bi	36'99	36'58	36'67
Cs	34'69	34'94	35'17
Cl	—	—	28'16

$3CsI \cdot 2BiI_3$. Cæsium-bismuth Iodide.

We could obtain only one double iodide of bismuth and cæsium, although the proportions of cæsium and bismuth were varied greatly. The salt formed as a crystalline precipitate, difficultly soluble especially in an excess of

cæsium iodide, when 1 grm. of bismuth iodide was added to 50 grms. of cæsium iodide, and when 1 grm. of cæsium iodide was added to 50 grms. of bismuth iodide. With an excess of cæsium, the colour was a bright red, while with an excess of bismuth the colour was more of a reddish brown.

Methods of Analysis.—The methods here given were used in both the double chlorides and iodide of bismuth.

Halogens were determined as the silver salts, being precipitated from a solution acidified with tartaric and nitric acids, and, after standing, filtered and weighed on a Gooch crucible. As Remsen and Brigham had mentioned a difficulty in determining bismuth, we made a few determinations of it in Bi_2O_3 , which was made by precipitating BiONO_3 with water from a nitric acid solution of $\text{Bi}(\text{NO}_3)_3$, and heating the precipitate to constant weight in a platinum dish. The method finally adopted was to dissolve the substance in water slightly acidified with hydrochloric acid and precipitate Bi_2S_3 from the cold solution with hydrogen sulphide. The precipitate was filtered and immediately dissolved in nitric acid and digested for some time on the water-bath until completely decomposed. The sulphur was filtered off and the filtrate, diluted to about 300–400 c.c., was heated and ammonium carbonate added in slight excess. It was placed on the water-bath for an hour or two, until the liquid had become nearly clear and the excess of ammonium carbonate had been driven off, and it was then filtered on a Gooch crucible and ignited strongly over a Bunsen burner and weighed as Bi_2O_3 .

Two determinations on Bi_2O_3 gave the following results:—

	Grm.		Grm.
I. Amt. Bi_2O_3 taken =	0.1979	Amt. Bi_2O_3 found =	0.1974
II. " " "	0.3604	" " "	0.3617

The filtrate from the bismuth precipitation was evaporated with sulphuric acid and ignited in a stream of air containing ammonia. The residue was weighed as Ca_2SO_4 .

The results obtained from the analysis of the double iodide were as follows:—

	I.	II.	Calculated for $\text{Ca}_2\text{Bi}_2\text{I}_6$.
Bi	21.34	21.15	21.25
Ca	20.75	20.31	20.38
I	—	58.02	58.37

—*American Journal of Science*, iii., No. 18, June, 1897.

PROCEEDINGS OF SOCIETIES.

CHEMICAL AND METALLURGICAL SOCIETY, JOHANNESBURG.

May 15, 1897.

AFTER a few remarks on Prof. Christy's paper, read at a previous meeting, the Society proceeded to discuss and comment on Mr. EHRMANN's paper on the "*Precipitation of Gold from Cyanide Solutions.*"

The drift of the discussion was as to the advantage, if any, compared with the increased cost, to be gained by heating the solution to promote rapid precipitation; it does not appear to be at all certain whether there is any appreciable quickening of the process during the summer months as compared with the winter, and, as is well known to those who have lived in the country, the range of temperature is very great. The cost of heating large quantities of solution would be very prohibitive.

A few remarks were made on Dr. STOCKHAUSEN's paper on "*The Liqumtion in Cyanide Bars,*" and Mr. D. J. WILLIAMS then read a few notes on "*The Estimation of Lead in Slags and other By-products.*"

SOCIÉTÉ D'ENCOURAGEMENT POUR L'INDUSTRIE NATIONALE.

June 25, 1897.

M. MASCART, President, in the chair.

AFTER reading the correspondence and hearing the reports of the committees on the Mechanical Arts and the Economic Arts a communication was made by M. CHEYSSON on the "*Insurance in French Industries against Accidents.*" The author was warmly thanked for his paper, which was ordered to be inserted in the *Bulletin*.

The President delivered a long discourse on the progress of the Society, and made special reference to absent and foreign members. A list of medals and prizes awarded was then read, and the meeting adjourned.

NOTICES OF BOOKS.

The Chlorination Process. By E. P. WILSON, E.M. First Edition, first thousand. New York: John Wiley and Sons. London: Chapman and Hall, Limited. 1897. Pp. v—125, 12mo., Ill.

PLATTNER laid the foundation for the process of extracting gold from its ores by the agency of chlorine, in 1856, and since then the method has undergone many improvements. The first to make a practical and commercial success of chlorination was Mr. G. F. Deitken, in California. Mears and Theis subsequently replaced tank-lixiviation by barrel-chlorination, and the latter method of treatment prevails.

Mr. Wilson has written very clearly and explicitly, showing familiarity with the literature of the subject, and with the practical workings of many American works. In successive chapters he describes the preparation of the ore, the methods of roasting, the furnaces, the leaching process, filtering methods, precipitation, and the refining of the precipitated gold; the final chapter is devoted to the cost of chlorination.

Mr. Wilson shows a great deal of common sense in his occasional remarks, as in the following paragraph:—"Some wise person has stated that 'all is not gold that glitters,' and, if he were alive and a miner, he could have added two other facts which history has established, viz., that all gold-bearing rocks do not contain gold in paying quantities; also, that some gold-bearing rocks contain considerable quantities of gold, but are commercially valueless."

Due consideration of these statements might have prevented great losses in gold-mining ventures.

Again he writes:—"The chlorination process has not been more generally adopted because those who run mines are not capable of carrying it on, and, knowing their weakness, let it alone. And parties having mining machinery to sell discourage the chlorination process. Lastly, mine-owners, thinking they will have to pay higher salaries to good men, are willing to suffer loss of gold rather than do so."

The book has been written in a style intelligible to mine-owners as well as to students of metallurgy, though the interests of the latter have been regarded by the introduction of chemical equations.

Mr. Wilson, in common with so many writers of English, uses the French word *résumé* where "summary" will serve equally well.

This little work forms a companion volume to the author's "*Cyanide Processes.*" The book is well printed on good paper, and contains an Index.

H. C. B.

The Agricultural Journal. Department of Agriculture, Cape of Good Hope. Vol. x., No. 10, May 13, 1897.

A GREAT deal of this number is taken up with matter purely of interest to farmers and stock-breeders; some attention is being paid to artificial manuring, as the farmers there are beginning to realise that, unless the production can be increased, farming will not pay, and that what is taken from the soil must be returned to it.

There are some long extracts from Dr. Wm. Newton's paper on Nitrates, read before the Liverpool Meeting of the British Association last year; and some further remarks by Dr. Dyer on the use of sulphate of ammonia for the same purpose.

In connection with the experiments being now conducted in the colony for the extirpation of *Tcerya Purchasi* (Australian bug) and scale insects, by the introduction of ladybirds from California and Australia, an instructive communication is produced, showing how entirely successful the same experiment has been in the Hawaiian Islands. The first importation was made in 1890, when *Vedalia cardinalis* was sent over, at the time when most trees were in a deplorable condition from the attacks of *Tcerya*; the *Vedalia* was a complete success. It became perfectly naturalised, increased prodigiously for a time, practically cleared the trees, and then—as the *Tcerya* became comparatively scarce—decreased in numbers, while at the present time it is evident that the number of the scale insect and its destroyer has arrived at a fixed proportion. The ladybird has previously done excellent service in the fruit orchards in Lower California. The effect is not imaginary, but proven. In June, 1895, a lovely forest in Hawaii—5000 feet above sea-level—was found to be much affected by a black *Aphis*. By beating the trees the blight came down in abundance. One or two introduced ladybirds were also noticed. By September the ladybirds were present in thousands,—the blight and native insects in small numbers. In August, 1896, not an *Aphis* was to be found, and only one or two stray ladybirds. They had done their work and disappeared.

Thirty-third Annual Report on Alkali, &c., Works. By THE CHIEF INSPECTOR. London: Eyre and Spottiswoode. 1897.

FOLLOWING the appointment of an additional Sub-inspector, there has been a re-distribution of district areas, which has relieved the work in some of the districts. The number of Works now registered in England, Ireland, and Wales under the Act is 1074. Of these 98 only are works decomposing salt, and so scheduled as Alkali Works, while the remainder, 976, carry on processes which are scheduled under the Acts of 1881 and 1892. These numbers show a decrease of three alkali works and an increase of twelve other works since 1895. There are also 125 works registered in Scotland, bringing the total number registered to 1199.

There has been one prosecution for "obstruction" under Section 17 of the Act of 1881. The important point was that, after the works had been passed for registration, on the basis of plans supplied by the proprietor, the manager had carried out the deception of the by-pass pipes, which prevented the Inspector from examining the effluent gases. The works in question, for the manufacture of carbon bisulphide, are now closed, and it is not likely the manufacture will be resumed. In addition, there were five cases of unregistered works being carried on; but in each case the fees were accepted by the Board, it appearing that ignorance existed of the obligations of the Act.

The amount of salt decomposed in the Leblanc process again shows a reduction, and one more considerable than has occurred since 1893. On the other hand, the ammonia-soda process has largely increased its lead over its rival, obtained for the first time last year.

A new departure in the manufacture of chlorate of soda, a salt much used by calico-printers, has taken place during the year, by the introduction, on the large scale, of Mr. J. Hargreaves's process for chlorinating hydrated sodium carbonate directly, in an absorbing tower, in which lixiviation of the products to remove the sodium chloride is also conducted.

There has been an increase in the amount of ammonia recovered and made, but it is anticipated that in a few years there will be a decrease, owing to the extensive adoption by gas engineers of carburetted water-gas plant in connection with gas manufacture in many parts of the country. In America coal-gas has been displaced to the extent of over 70 per cent by carburetted water-gas; in this manufacture no ammonia is produced.

General Index to the Proceedings of the Society of Public Analysts. Compiled by J. C. WELCH, F.C.S. London: Baillière, Tindall, and Cox. 1897.

THE compiler originally commenced this work for his own private convenience, but the allusion to this subject made by Mr. Otto Hehner, in his Presidential Address in 1893, led him to offer the manuscript to the Society when completed to the end of the twentieth volume.

The thanks of public analysts generally are due to Mr. Welch for the care he has taken and the time he has expended in the laborious task.

CORRESPONDENCE.

THE PREPARATION OF ZINC ETHYL.

To the Editor of the Chemical News.

SIR,—In the last issue of the CHEMICAL NEWS (vol. lxxvi., p. 20) there appears under this heading an abstract of a paper by A. Lachman from the *American Chemical Journal*. Mr. Lachman employs a copper-zinc couple, which he prepares by mixing zinc-dust with copper oxide, and then reducing the copper by passing hydrogen over the heated mixture. For many years I have been in the habit of preparing zinc ethyl, using a copper-zinc couple consisting of a mixture of zinc dust and copper oxide, *without reducing the copper oxide to the metallic state*, and a description of the process was published in my "Chemical Lecture Experiments" in 1892. Such a mixture acts upon ethyl iodide with extreme readiness; I have performed the entire operation of preparing zinc ethyl and distilling it off during a lecture of an hour.—I am, &c.,

G. S. NEWTH.

Royal College of Science, London.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 24, June 14, 1897.

Contribution to the History of the Phosphorus Iodides.—A. Besson.—The solution appears to contain an instable compound, P_2I_4 , which is the pivot of the apparent transformation of white phosphorus into red phosphorus by the action of iodine.

Procedure of Oxidation and Chloridation.—A. Villiers.—When an oxidisable body occurs in a medium capable of furnishing oxygen, but under such conditions

that the oxidation either does not begin at all or takes place very slowly, the addition of a trace of a salt of manganese determines the reaction or accelerates it considerably.

Splitting up of the Fundamental Band of the Chlorophylls.—A. Etard.—The number of chlorophyll bands and the wave-length of their mean axis may, by the method of limited dilutions, be counted exactly, and may serve to characterise the chemical species. The diversity of the chlorophylls is demonstrated by the wave-length of the axes of their bands, whether pre-existing or induced by the action of reagents. The fundamental band of the chlorophylls is not always uniformly obscure; it may be double or triple.

On the Oxidising Action of Manganese Salts, and on the Chemical Constitution of the Oxidases.—G. Bertrand.—All the manganous salts which the author has tried possess the property of fixing free oxygen upon hydroquinone. They behave in the same manner with pyrogallol, paramidophenol, and other kindred bodies.

Action of Nickel upon Ethylene. Synthesis of Ethane.—Paul Sabatier and J. B. Senderens.—If we direct a mixture of equal vols. of ethylene and hydrogen upon nickel recently reduced and slightly heated (30° to 45°), we observe a notable rise of temperature, owing to the formation of ethane; copper, iron, and cobalt cannot serve to effect this synthesis.

Isolauronic Acid.—G. Blanc.—This memoir will be inserted at length if possible.

Action of Acetylene upon Silver Nitrate.—R. Chavastelon.— C_2H_2 forms with silver nitrate, according to the nature of the solvent, $C_2Ag_2.NO_3Ag - C_2Ag_2$. In future communications the author will describe a procedure for the determination of acetylene applicable in a great number of cases. He will also study the crystalline compounds of acetylene with cuprous chloride and mercuric chloride.

Determination of Resin Oil in Oil of Turpentine.—A. Aignon.—The author gives his results in the form of a table. It is possible, even on rectifying at 100° under a pressure reduced to 0.06 metre, to obtain a dextro-rotatory residue. The pure oil, if treated in the same manner, gives only lævo-rotatory residues.

Active Principles of some Aroids.—Mlle. Chau-liaguet, A. Hébert, and F. Hein.—The authors have not been able to detect the presence of hydrocyanic acid. The symptoms of poisoning by arums and the appearances observed on autopsy do not resemble those occasioned by hydrocyanic acid.

Action of Albumoses and Peptones on Inter-vascular Injection.—E. Fiquet.—The author's results are interesting physiologically rather than chemically.

Revue Universelle des Mines et de la Metallurgie.
Series 3, Vol. xxxviii., No. 2.

This issue contains no matter of chemical interest.

Revue Générale des Sciences Pures et Appliquées.
No. 10, May 30, 1897.

This number contains no original matter of special chemical interest.

Journal de Pharmacie et Chimie.
Series 6, vol v., No. 11.

Contribution to the Study of the Preparation of Ordinary Ether.—L. Prunier.—In the study of the preparation of ordinary ether by means of sulphuric acid and alcohol, most workers have omitted to take notice of the presence of sulphonic acids and their derivatives. This group of bodies is, however, to be found in notable

quantities in commercial ethers. It is also found in considerable proportions in the oils which have been used in the rectification of the raw product. By direct experiment it is possible to prove the formation of several sulphonic derivatives, especially towards the end of the operation. To separate the derivatives actually formed by the action of sulphuric acid, it suffices to heat sulphovinic acid with dilute sulphuric acid to 140°, then add a little alcohol. By this means a small quantity of ordinary ether is formed, and several sulphonic derivatives of varying volatilities, some of which even distil over with the ether. They are formed in greatest abundance when the temperature exceeds 140°, and above all if undiluted sulphuric acid be used. It is exactly the same as in the preparation of ethylene; if the operation be interrupted when the liquid becomes dark (165° to 175°) we can at that moment detect the presence of a large quantity of sulphonic compounds which exist in the liquid, along with the sulphuric acid in excess, traces of sulphovinic acid neutral sulphuric ether, polyethylenic compounds, and also sulphurous acid.

Researches on the Composition of Extracts of Meat.—J. Bruylants.—It has long been admitted that extracts of meat contain, as proteic substances, but a small quantity of gelatin. A large proportion of the nitrogen belongs to other proteic substances, albumoses, and peptones; that is to say, to substances whose nutritive value is greater than that of albumides, since they have already undergone some of the modifications due to digestion. The result of the examination of a number of different samples of extracts shows that Liebig's extract is the richest in the most nutrient and assimilable substances, though dry Bovril is not far below it; liquid Bovril is comparatively poor.

Method of Estimating Aldehyd in Ether.—L. Francois.—Already inserted.

Different varieties of Chestnuts.—M. Balland.—Not suitable for abstraction.

Reaction enabling Naphthol- α to be easily Distinguished from Naphthol- β .—E. Léger.—On April 7th the author made a communication on the action of hypobromite of sodium on certain phenols. In view of the facts therein described, he prepares from the supposed mixture a saturated aqueous solution; this solution, when diluted with its own volume of water, will not give the naphthol- β reaction with 2 drops of hypobromite, but if the substance under examination contain naphthol- α we get a violet or violet-rose colouration. By this means it is easy to detect 1 part of naphthol- α in 100 parts of naphthol- β .

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xvii.-xviii., No. 11. June 5, 1897.

Action of Hydrate of Chloral on Phenylhydrazine. Diphenylglyoxazol and its Derivatives.—H. M. Causse.—Hydrate of chloral unites very easily with phenylhydrazine, forming as a rule a crystalline compound, but sometimes an oil denser than water is produced; the crystals, however, are very unstable, so much so that analysis is impossible, the spontaneous decomposition being so rapid. The author has therefore been obliged to pass over the intermediary compounds and confine his research to the final products.

Monobromated Camphor.—Ch. Moureu.—Monobromated camphor is possessed of a remarkable stability; the brown appears to be united to the carbon as firmly as in aromatic bodies. Heated to 200° with phosphoric anhydride, an energetic reaction takes place, tarry products are formed, an abundance of gas is given off, and a liquid—fuming very strongly in contact with air—is distilled over. This latter has been found to be tribromide of phosphorus, the formation of which is of great interest.

MISCELLANEOUS.

Iron and Steel Institute.—The autumn meeting of the Iron and Steel Institute will be held at Cardiff, under the presidency of Edward Martin, Esq., on the 3rd, 4th, 5th, and 6th of August next. The following papers will be read, and members wishing to take part in the discussions will, on application to the Secretary, Mr. B. H. Brough, have copies of the papers forwarded to them a week in advance, as far as that may be possible:—

- "On Passive Iron." By J. S. de Benneville (Philadelphia).
- "On the Diffusion of Sulphides through Steel." By E. D. Campbell (Ann Arbor, Michigan).
- "On the Manufacture of Tin Plates." By George B. Hammond (Penarth).
- "On a Spectroscopic Analysis of Iron Ores." By Prof. W. N. Hartley, F.R.S., and Hugh Ramage, Assoc. R.C.Sc.I., F.I.C. (Royal College of Science, Dublin).
- "On Improvements in Shipping Appliances in the Bristol Channel." By Sir W. T. Lewis, Bart. Member of Council.
- "On the Iron Industry of Hungary." By D. A. Louis, F.I.C. (London).
- "On a Thermo-chemical Study of the Refining of Iron." By Prof. Honoré Ponthière (Louvain).
- "On Carbon and Iron." By E. H. Saniter (Wigan).
- "On some Mechanical Appliances at Penarth Docks." By T. Hurry Riches, M.Inst.C.E. (Cardiff).
- "On the Application of Travelling Belts to the Shipment of Coal." By Thomas Wrightson, M.Inst.C.E. (Thornaby-on-Tees).

There will be, as usual, a number of excursions to works and other places of interest in the neighbourhood, particulars of which, together with the full programme of the meeting, may be obtained from the Secretary, at 28, Victoria Street, London, S.W.

Combustion of Nitrogen.—O. Bleier (*Berichte*).—Nitrogen is mixed with oxygen in the proper proportion, introduced into an enamelled autoclave, or other suitable vessel, containing dilute alkali, and an excess of detonating gas is pumped in. The mixture is exploded, and the oxides of nitrogen are removed by shaking up with alkali. Detonating gas is again introduced, and the process is repeated.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 5th inst., Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, presiding. The following were elected Members:—H. H. Baird, Ivon Braby, J. M. Davidson, M.B., C.M., A. C. Hill, B.A., J. Y. Johnson, L. Kamm, M. E. Stephens, The Rev. Henry Wace, D.D., Julius Wernher, and Henry Wilde, F.R.S. The Special Thanks of the Members were returned to Sir Andrew Noble for his donation of £100 to the Fund for the Promotion of Experimental Research at Low Temperatures.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Marking Inks.—(Reply to "Sulpho").—Ample information upon the subject, with the latest recipes, will be found in the Second Series of Spon's "Workshop Receipts," 1890.—GEORGE TURNER, 5, South Street, E.O.

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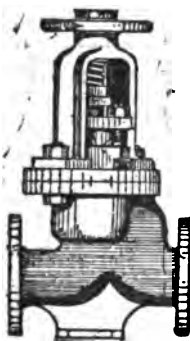
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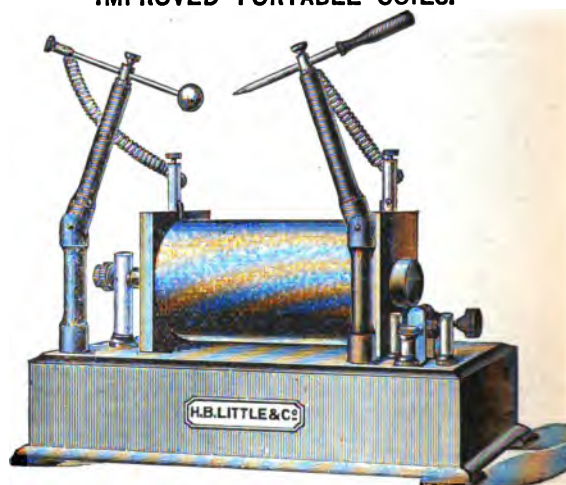
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Vol. LXXVI., No. 1965.

MIGRANT MATTER.

By STEPHEN H. EMMENS and NEWTON W. EMMENS.

On the 5th of March, in this year, we cut a circular disc, 1 inch in diameter, from a thin sheet of lead purchased as being "chemically pure" and showing no residual metal on cupellation. We also cut a similar disc from a thin sheet of silver purchased as being "chemically pure." These discs were then weighed and placed in contact with the two ends of a short spiral of copper wire of known weight; and the system thus composed was suspended inside a wide-mouthed glass bottle by means of a string depending from the cork and passing through the spiral and central holes in the discs. The silver disc formed the lowest member of the system.

On the 15th of March the bottle, which had meanwhile stood on one of the shelves in our laboratory, was opened, and the discs and spiral were taken apart and weighed. They were then replaced as at first, and allowed to remain untouched until the 2nd inst., when they were again weighed.

The figures (in grms.) of the three weighings were as follows:—

	Lead.	Silver.	Copper.
March 5 ..	0.8931	0.50845	1.5421
" 15 ..	0.8932	0.50830	1.5420
June 2 ..	0.8932	0.50810	1.5421

We dissolved the copper in nitric acid. The solution, on being tested with hydrochloric acid, gave no reaction for silver. But the lead disc, on cupellation, gave a silver bead weighing 0.00003 grm.

It would appear, from this experiment, that what is commonly recognised as solid silver is, in part at least, a migrant mode of matter. It would also appear possible that the gold found by Prof. Roberts-Austen in cylinders of lead which had been standing for some time upon golden bases may have migrated from the outer surface of such bases instead of travelling through the interior of the metallic column. The atmosphere certainly seems to be the path of least resistance.

We use the term "migrant matter" because the travelling particles to which we refer are (in common with odours generally) much more akin to Crookes's "fourth form" than to gases.

Argentaurum Laboratory,
June 5, 1897.

ON THE ESTIMATION OF OXYGEN DISSOLVED IN SEA-WATER.

By ALBERT LÉVY and F. MARBOUTIN.

SOME time ago one of us published the details of a precise and rapid method of estimating the oxygen dissolved in waters. It having been in constant use for fifteen years at the Observatory at Montsouris, we have been able to follow, week by week, the variations existing, from the point of view of oxygen dissolved, in waters from springs, rivers, and drainage. The method consists in partially peroxidising, by the help of the oxygen dissolved in the water, an excess of protoxide of iron, and completing the oxidation by means of a titrated solution of permanganate

of potash. We have described in the different *Annals* of Montsouris the method of procedure, and have each year discussed the results obtained. This method, however, presented several difficulties when we wished to apply it to the analysis of waters containing much chlorides, such as sea-water. In this case, at the moment of adding the permanganate, we observe a disengagement of chlorine which conduces to a too high reading:—

50 c.c. fresh water, reading 22.80 c.c. No chlorine.
50 c.c. fresh water + 50 c.c. of chloride solution, reading 23.00 c.c. Cl given off.
50 c.c. fresh water + 100 c.c. of chloride solution, reading 23.30 c.c. Cl given off.

We can, however, even with waters high in chlorides, succeed in getting good results with permanganate, by making a special datum of comparison for each water and agitating the liquid cautiously, and always working in an identical manner.

But we prefer, for such waters high in chlorides, to replace the permanganate by bichromate of potash, and to determine the end of the operation, by means of the touch test, with ferrocyanide of potassium. We have made the following observations:—

1. In the case of a spring or river water, the permanganate and the bichromate give precisely the same result, and this result is identical with that obtained by extracting the gas with a mercury pump.

Eau d'Aure, taken 10th March, 1897, from the Reservoir in the Rue Villejust.

Permanganate method: volume of water 95.2 c.c.
Datum .. 24.00 c.c. Reading .. 17.30 c.c.

In 1 litre of water,—
 $\frac{24.00 - 17.30}{95.2} \times 160.8 \dots 11.32 \text{ m.grms.}$

Bichromate method: volume of water 96.6 c.c.
Datum .. 23.80 c.c. Reading .. 17.00 c.c.
In 1 litre of water,—
 $\frac{23.80 - 17.00}{96.6} \times 160.0 \dots 11.26 \text{ m.grms.}$

Oxygen extracted by the mercury pump:—Amount operated on, 364.8 c.c. Oxygen measured at 0° C. and 760 m.m., 2.872 c.c.

In 1 litre of water,—
 $1.43 \times \frac{2.873 \times 1000}{364.8} \dots 11.26 \text{ m.grms.}$

2. The oxygen dissolved in sea-water is estimated very exactly by the use of bichromate, in spite of the large quantity of chlorides and magnesian salts.

We worked on a sample taken in a carboy, sent from Concarneau by the kindness of M. Fabre-Domergue.

Three determinations by means of bichromate gave, per litre of water,—

1st. $\frac{23.50 - 17.40}{102.6} \times 160.0 \dots 9.52 \text{ m.grms.}$

2nd. $\frac{23.50 - 17.65}{98.3} \times 160.0 \dots 9.52 \text{ m.grms.}$

3rd. $\frac{23.50 - 17.75}{96.6} \times 160.0 \dots 9.53 \text{ m.grms.}$

Oxygen extracted with the mercury pump: Amount operated on, 364.800 c.c. Oxygen measured at 0° C. and 760 m.m., 2.432 c.c.

In 1 litre of water,—
 $1.43 \times \frac{2.432 \times 1000}{364.8} \dots 9.51 \text{ m.grms.}$

When waters such as sea-water contain much magnesia, at the moment, following our method, when we make the liquid alkaline with potash, we see the magnesia precipitated in the form of little cylinders similar to grains

of rice. This precipitate in no way interferes with the success of the operation; it is only necessary to turn the tube holding the liquid up and down a few times, so that the precipitate may be equally disseminated throughout the solution.

Bichromate retains its titration almost indefinitely. —*Bull. Soc. Chim.*, Series 3, vol. xvii.-xviii., No. 12.

ON THE VOLUMETRIC DETERMINATION OF ZINC BY POTASSIUM FERROCYANIDE.

By L. L. DE KONINCK and EUG. PROST.

(Continued from p. 30).

THE two series were done at an interval of several days, and with different ferrocyanide solutions; that used for the first series (Solution A) was prepared from salt purified by crystallisation, the other (Solution B) from ordinary commercial ferrocyanide. It may be noted that the figures of the second series diminish more regularly than those of the first; that is caused by the reason, that in the second series the titration back was not done until after digestion for from twenty to forty-five minutes; while in the first, no account was kept of the time—its influence on the result being not then known to us.

One sees that the influence of chloride of ammonium is very marked; the result differs considerably according to whether the solution does (Nos. 3 to 8) or does not (Nos. 1 and 2) contain any, but within the ordinary limits of practical working (Nos. 3 and 4) there does not seem to be much influence. It will evidently be necessary in making estimations in the presence of chloride of ammonium to work in such a manner that the quantity of this salt should be as near as possible constant, and to titrate the ferrocyanide in presence of a similar quantity.

We are unable to find any explanation of the influence of chloride of ammonium in showing a higher relation between the ferrocyanide and the zinc. At first sight one might think, as with hydrochloric acid, that ferrocyanide being slightly soluble in the reagent in question—in this case a salt of ammonia—an excess of ferrocyanide would be necessary to produce a complete precipitation; but this is not so, as a very simple experiment shows.

By dropping 2 drops of $\frac{1}{2}$ normal ferrocyanide into 15 c.c. of water to which has been added 1 drop of a $\frac{1}{2}$ th normal solution of neutral zinc salt, a hardly noticeable cloudiness is produced, while if we repeat the experiment, but substitute a 20 per cent solution of ammonium chloride for the water, we get a very distinct cloudiness. On the other hand, if we divide the liquid of the first experiment into two equal parts, then add to one 10 c.c. of water, and to the other 10 c.c. of 20 per cent ammonium chloride, we notice that the latter becomes distinctly cloudy, while the former shows no change. Thus, chloride of ammonium favours precipitation.

The end of the reaction is very distinctly marked, especially in Experiments 3 to 6.

Influence of Nitrate of Ammonium.—In the analysis of minerals one is liable to have a small quantity of nitrate of ammonia present, from the fact of using nitric acid in dissolving the sample or for the oxidation of ferrous salts. We thought it advisable to ascertain the influence of this salt.

The experiments were carried out by using 20 c.c. of $\frac{1}{2}$ normal ZnCl_2 , 50 c.c. of $\frac{1}{2}$ normal A solution of ferrocyanide, 10 c.c. of 5 normal HCl, 50 c.c. of 20 per cent AmCl , and 100 c.c. of water, and titrating back with chloride of zinc after digestion for fifteen minutes.

	AmNO_3 .	ZnCl_2 back.	ZnCl_2 total.
1	0 grm.	4'55	24'55
2	2 grms.	4'55	24'55
3	5 "	4'55	24'55

The end of the experiment was perfectly clearly marked. We see thus that nitrate of ammonia is without any influence whatever within these limits, and we did not think it worth while pushing the experiments any further.

Influence of Hydrochloric Acid.—In these experiments we used 20 c.c. of $\frac{1}{2}$ normal ZnCl_2 , 50 c.c. of $\frac{1}{2}$ normal ferrocyanide, 50 c.c. of 20 per cent AmCl , and 110 c.c. of water, and 5 normal hydrochloric acid in the proportions shown below:—

	5 normal HCl.	Water.	ZnCl_2 back.		ZnCl_2 total.	
			I.	II.	I.	II.
1. .. 10	100	5'00	4'85	25'00	24'85	
2. .. 20	90	4'80	4'71	24'80	24'71	
3. .. 30	80	4'75	4'56	24'75	24'56	
4. .. —	—	4'60	—	24'60	—	
5. .. 40	70	4'40	4'46	24'40	24'46	
6. .. 50	60	4'20	4'21	24'20	24'21	
7. .. —	—	4'40	—	24'40	—	
8. .. 60	50	4'05	3'91	24'05	23'91	
9. .. 70	40	3'75	3'72	23'75	23'72	

As in the case of the experiments with chloride of ammonium, we again notice the difference of regularity between the first and second series; the reason of this difference is the same as before; the back titration of the second series was not done until after digesting for twenty minutes in the case of No. 1, to forty minutes for No. 9; while in the first series the back titration was done immediately, but without taking any note of the time elapsing between the addition of the ferrocyanide and the titrating back.

It is obvious that the influence of hydrochloric acid is similar to that of chloride of ammonium; the greater the acidity, the more ferrocyanide there is required to completely precipitate the zinc, or at any rate to show with the indicator.

As we have already remarked with regard to ammonium chloride, it is necessary, in order to obtain very exact results in the estimation of zinc, to use a solution of as near as possible constant acidity for the estimations themselves, as well as in the titration of the ferrocyanide.

Influence of Sulphurous Acid.—A freshly-prepared solution of ferrocyanide of potassium has hardly any colour, but after some time it assumes a yellowish tint. When we make an estimation of zinc with such a solution, we note—after the precipitate has entirely settled—that the liquid also has a distinct yellow colour. This colouration, which Galletti attributed to the solution of zinc ferrocyanide in the acid liquor, is due to ferricyanide. This is easily shown by means of ferric chloride. When, in the analysis of a mineral, it is necessary to treat the solution with hydrosulphuric acid, so as to precipitate the copper, cadmium, &c., we ought to re-oxidise the salts of iron with nitric acid, or bromine, and then only precipitate the iron with ammonia.

When, in our first experiments on minerals, of which we shall speak later on, we used nitric acid for re-oxidation, in the ordinary way, we found that the resulting solution had, after the precipitate had settled, a yellow colouration, considerably more intense than in the case of minerals which had not been subjected to re-oxidation. We were easily enabled to prove that this phenomenon was due to the oxidation of a part of the ferrocyanide by the nitrous acid (Schaffer's reaction, *Sill. Amer. Journ.*, Series 2, vol. xii., p. 117, 1854; see also Van Deventer, *Ber.*, xxvi., p. 589, 1893; and Van Deventer and Jorgens *ibid.*, 932). This latter results from the nitrite of ammonium formed by the action of ammonia on the nitrous compounds produced by the oxidation of the ferrous salts by nitric acid. The ammoniacal filtrate separated from the ferric hydrate precipitate contains a good amount of nitrite, for if we add a little chloride of potassium and then acidulate the solution it shows a distinct colour from the iodine set free.

Similar phenomena to those we have just described are—

noticed if we use an excess of bromine to oxidise the ferrous salts, and then precipitate the manganese with the iron by means of ammonia. By the action of the ammonia on the excess of bromine, there is, without doubt, a little hypobromite or bromate of ammonia produced; for on making the liquid acid, we can detect the presence of a small quantity of free bromine, and if we titrate with ferrocyanide under these conditions we note also the formation of ferricyanide. We can, however, get over these difficulties by adding a small quantity of sulphite of soda to the ammoniacal solution before making it acid. Solutions treated in this manner are, after titration and when the precipitate has settled, absolutely colourless.

Before recommending the use of sulphite of soda, we thought it necessary to assure ourselves that it was really innocuous. That we did in the following manner:—We used 20 c.c. of $\frac{1}{2}$ normal ZnCl_2 , 100 c.c. of water, and 15 c.c. of 5 normal HCl. We then added 50 c.c. of $\frac{1}{2}$ normal ferrocyanide (A), and after digesting for fifteen or twenty minutes, we titrated back with zincic chloride.

A. Without the addition of sulphite of soda:—

1. ZnCl_2 back	4.65 c.c.	ZnCl_2 total	24.65
2. " "	4.62 " "	" "	24.62

B. With the addition of 10 c.c. of normal sulphite of soda (12.5 grms. $\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$ per litre) to the zincic solution:—

3. ZnCl_2 back	4.69 c.c.	ZnCl_2 total	24.69
4. " "	4.70 " "	" "	24.70

The solutions, after the sulphide was added, smelt strongly of sulphurous anhydride. The end of the reaction showed more slowly, but as distinctly, with sulphurised solutions as with others, only the brown colouration of ferrocyanide of uranium is less distinct, and, in the case of weak solutions, disappears after a few minutes. It must be remembered that the quantity of sulphite here used is considerable, and corresponds molecularly to the 20 c.c. of $\frac{1}{2}$ normal ZnCl_2 on which the experiments were made, while in ordinary estimations 0.1 to 0.2 c.c. of normal sulphite would be amply sufficient. It is evident that in this proportion its action on the indicators would be nil, as we have conclusively shown.

Influence of Bromine.—Although it would appear evident *a priori* that the adverse influence of the excess of bromine used to oxidise the ferrous salts might be neutralised by means of sulphurous acid, we thought it well to verify the fact by experiment.

1. 20 c.c. of $\frac{1}{2}$ normal ZnCl_2 were made alkaline with 10 c.c. of commercial ammonia, then acidulated with 30 c.c. of 5 normal HCl. We then added 100 c.c. of water, and ran in 50 c.c. of $\frac{1}{2}$ normal ferrocyanide.

2. To 20 c.c. of $\frac{1}{2}$ normal ZnCl_2 we added 15 c.c. of water saturated with bromine, then treated it with ammonia, and acidulated with 30 c.c. of acid; the solution turned a deep yellow. The bulk of the bromine was driven off by boiling, the last traces were got rid of by a few drops of normal sulphite of soda, the liquid was cooled, and 50 c.c. of $\frac{1}{2}$ normal ferrocyanide were added. The back titration with chloride of zinc gave—

1. ZnCl_2 back	4.15 c.c.	ZnCl_2 total	24.45
2. " "	4.50 " "	" "	24.50

The use of bromine has therefore no influence, or rather it is neutralised by the use of sulphite of soda.

Influence of Nitric Acid.—The following experiments were carried out with the object of satisfying ourselves that the addition of sulphite of soda to the zincic solution would neutralise the action of the nitrous acid, which is formed, under the conditions we have already stated, by the oxidation of the ferrous salts with nitric acid. A cuprous and ferruginous calamine, of which five assays were made, either with sodic sulphide or with ferrocyanide and sodic sulphite, showed a proportion of 23.94 to 24.39 per cent of zinc. After treatment with hydrosulphuric

acid and re-oxidation of the salts of iron by nitric acid it gave the following results:—In a preliminary experiment in duplicate, 30.84 per cent Zn; in a second 31.32 and 31.48 per cent Zn—either an error of more than 7 per cent of the mineral or of nearly 29 per cent of the Zn present. The solutions, after the deposition of the precipitate, were of a very deep yellow colour. The concordance of the results obtained with this sample of ore, on the one hand with sulphite of sodium, and on the other with ferrocyanide and the addition of sulphite, shows that the influence of the nitrite can be entirely done away with by this latter reagent. We, however, made a special experiment to prove it.

We used a 5 normal solution of nitrite of soda (6.9 grms. per litre) and a normal solution of sulphite of soda in the proportions given below.

The solutions were prepared as follows:—To 30 c.c. of $\frac{1}{2}$ normal zincic chloride we added the sulphite of soda, the nitrite diluted to 100 c.c. with water, 10 c.c. of 20 per cent chloride of ammonium, 10 c.c. of 5 normal hydrochloric acid, and 35 c.c. of ferrocyanide of potassium approximately $\frac{1}{2}$ normal for zinc. After standing not less than twenty minutes, we titrated back with the zincic solution.

	NaNO_2 $\frac{1}{2}$ normal.	Na_2SO_3 Normal.	ZnCl_2 $\frac{1}{2}$ normal.
1.	—	0.5 c.c.	3.94 c.c.
2.	1 c.c.	1.0	3.93
3.	2	2.0	3.90
4.	5	3.0	3.88
5.	10	5.0	3.90

The use of sulphite thus neutralises the action of the nitrite absolutely; the final solution is quite colourless.

Influence of Manganese.—Manganese, which is sometimes found, though in small quantities only, in zinc ores, requires special care in its elimination. It has been thought that in acid solutions it was not precipitated by ferrocyanide, and was therefore without influence.

A quantitative experiment is sufficient to show that in order to prevent the precipitation of manganous ferrocyanide there must be present such a proportion acid as to seriously influence the precipitation of the zinc itself. However, we made a definite experiment. We used 20 c.c. of $\frac{1}{2}$ normal ZnCl_2 , 50 c.c. of $\frac{1}{2}$ normal ferrocyanide, 50 c.c. of 20 per cent AmCl , 10 c.c. of 5 normal HCl, and 100 c.c. of water, and digested for fifteen minutes.

1. Without manganese		ZnCl_2 back	4.50 c.c.
2. With 0.0275 gm. manganese	" "	" "	2.27 "

The manganese thus acted on the ferrocyanide the same as 2.23 c.c. of $\frac{1}{2}$ normal ZnCl_2 . The quantity of manganese added was equal to 2 c.c. of $\frac{1}{2}$ normal solution, and one sees that this metal requires for its precipitation at least as much ferrocyanide as zinc does. Manganese must therefore be entirely eliminated before titration.

(To be continued).

THE SOLENOID ELECTRO-MAGNET.

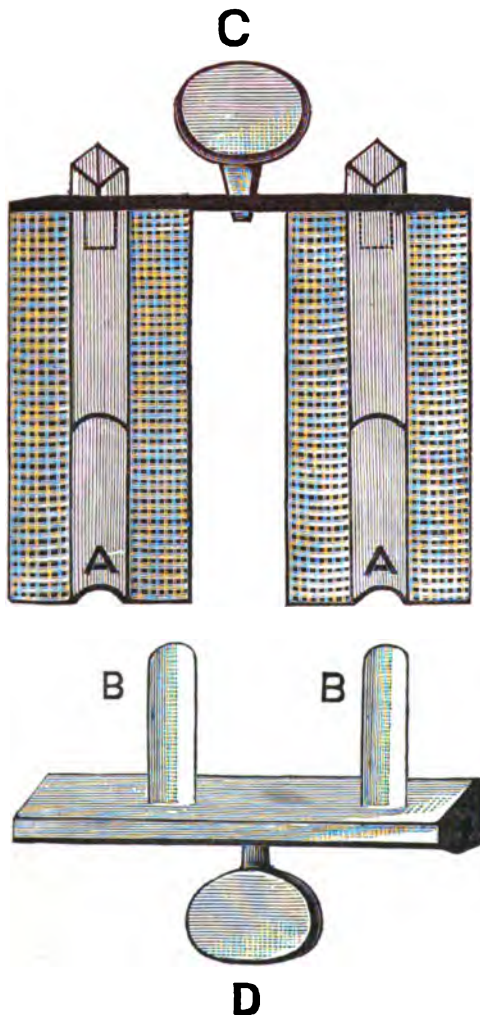
By H. N. WARREN, Principal, Liverpool Research Laboratory.

THIS powerful type of electro-magnets, which after a lengthy and exhaustive research, has just been perfected at the Research Laboratory, differs from all other magnets in the construction of the iron core; they being intended for either supporting great weights in general, or the accommodation of spherical bodies, for which reception a hollow cavity is cut from each extremity, the section of each cavity representing a semicircle.

For the construction of the iron core, iron oxides, free from sulphides, silicates, and carbonaceous matter, were selected, and reduced in an atmosphere of hydrogen,

puddled through a hot-blast Siemens furnace, and drawn into bars; the bars are next imbedded in quicklime, brought to a full white heat, and allowed to cool in that substance. After cooling, each limb before yoking is drilled to within half an inch of its circumference and one-quarter of its total length (if the bar be less than half inch from starting, a corresponding allowance has naturally to be made).

The following diagram illustrates the section of a magnet with its accompanying keeper, the separate parts being further described in the specified table. The magnet was constructed to suspend a weight of 10 tons, and required to be excited by a current obtainable from 25 boron carbon cells only.



From the above diagram it will be readily observed that when the keeper is applied with its two iron projections, B B, which are constructed of such dimensions as to exactly coincide with the openings A A; not only is the magnetic power fully utilised, but any side-slipping of the keeper is absolutely impossible. All the magnets thus described were wound with No. 14 double cotton-covered wire, the winding afterwards being covered with paraffined insulation, wound with thread, and varnished with best electro shellac varnish: they are at present doing excellent service in researches on diamagnetism and polarisation of light.

The subjoined table will better explain a few of the more important magnets thus constructed, and giving approximate quantities of their component parts:—

Length and diameter of core.	Weight of primary.	Voltage required.	Weight supported.
2 inches $\times$ $\frac{1}{4}$	4 ozs.	6	8 lbs.
6 " $\times$ $\frac{1}{4}$	1 lb.	10	80 "
28 " $\times$ 2	9 lbs.	50	5 cwts.
28 " $\times$ 4	100 "	50	2 tons.
36 " $\times$ 4 $\frac{1}{2}$	112 "	50	10 "

18, Albion Street, Everton, Liverpool.
Liverpool Research Laboratory,

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING JUNE 30TH, 1897.

By WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, *Metropolis Water Act*, 1871.

London, July 10th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 168 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from June 1st to June 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in a previous report.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 168 samples examined all were found to be clear, bright, and well filtered.

The rainfall at Oxford during June was 1.7 inches; the average for the last 30 years is 2.68 inches; this leaves a deficiency of 0.98 inch on the month, making an actual excess of 0.06 inch for the year, on a fall of 11.37 inches.

The results of our bacteriological examinations of 230 samples are recorded in the following table; we have also examined and reported on 34 other samples taken at various places, such as stand-pipes, and different filter-beds:—

	Microbes per c.c.
Thames water, unfiltered (mean of 24 samples)	73893
Thames water, from the clear water wells of five Thames-derived supplies (mean of 112 samples)	74
Ditto ditto highest	558
Ditto ditto lowest	6
New River, unfiltered (mean of 24 samples)	2005
New River, filtered (mean of 23 samples)	69
River Lea, unfiltered (mean of 24 samples)	66822
River Lea, from the clear water well of the East London Water Company (mean of 23 samples)	69

The remarks made last month as to the impossibility of comparing microbial estimations on the same water by different experimenters unless the samples were taken at the same time and place, and the subsequent processes

of treating the samples for plate cultivation of bacteria were absolutely identical, are strikingly borne out by a report recently addressed to the London County Council by their chemist, where stress is laid on a great discrepancy between the numbers given by Sir Edward Frankland and ourselves.

A discussion of differences of this kind will only be possible when all the analysts adopt the same method of bacteriological cultivation.

Samples of raw river water show continual variations according to the season, atmospheric conditions, and rainfall. For instance, on June 8th we found in the Thames, 270,320 microbes per c.c., and on the 9th 9920. On the 10th we obtained 982,080, and on the 11th 58,640. In the river Lea on June 14th we found over 1,500,000 microbes per c.c.: on the 16th, 240; on the 17th, 120; and on the 18th, 9920.

We take several samples daily and give the averages. Other chemists taking samples once a week or once a month could not, unless by mere chance, hit upon the averages we record.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.
JAMES DEWAR.

NOTES ON LUCIUM.

By WALDRON SHAPLEIGH.

IN May of last year I secured several samples of the so-called "lucium" which had been prepared by Dr. Leon Schützenberger, of Paris, and at the same time saw a report in which this chemist gave very fully the methods which he used in preparing it, and his conclusions regarding the same, also samples of the monazite sand used.

The methods he employed follow very closely those set forth in the patent of Mr. P. Barrière.

The monazite sand was a low-grade North Carolina sand, containing minerals rich in titanium, zirconium, and yttrite earths, such as menaccanite, rutile, zircon crystals, samarskite, euxenite, and xenotime, and it is probable that from the last three minerals the "lucium" is obtained.

On experimenting with a solution of the lucium nitrate the ignited ash or residue I found to be of a dirty grey colour, and nearly entirely soluble in hydrochloric and nitric acids even when diluted. This solution was clear and pink, and gave the absorption bands of erbium. As the time recommended by Mr. Barrière for digesting with the potassium sulphate for the separation of the cerite group seemed to be rather short, I saturated the solution with potassium sulphate in the cold and allowed it to stand forty-eight hours, thereby obtaining a precipitate consisting of the cerite group, and in the solution the yttrite group. Analysis showed:—

Yttrite group oxides	93.98
Lanthanum, cerium, didymium oxides ..	3.74
Thorium oxide	1.07
Titanium, zirconium, potassium, sodium oxides, and sulphuric acid, not separated	1.21
	100.00

The oxides of the yttrite group after this thorough purification by potassium sulphate were of a light buff or yellow colour, fully and completely soluble in dilute acids. Sodium hyposulphite did not give a precipitation in a fairly concentrated solution, nor was there any difficulty in making a partial separation of the erbium nitrate in the fused nitrates by Bahr and Bunsen's method. Although the quantity at my disposal was small, I obtained the erbium

nitrate crystals and showed the possibility of this separation.

Regarding the precipitation with sodium hyposulphite, on which great stress is laid as a reaction, showing "lucium" to be a new element, if a solution of yttrite sulphates in a concentrated solution of potassium sulphate made by cold digestion is heated, a copious precipitation will ensue. Though not a perfect separation, yet advantage can be taken of this reaction in separating yttrium from erbium, and this reaction should be borne in mind when separating the cerite from the yttrite group, as some text-books recommend using hot saturated solutions. By doing so, some of the yttrium will be precipitated with the cerite group.

This action of heat on the double sulphate solution of the yttrite group, if it does form similar salts to the cerite group, I have not met with in the text-books. Therefore, inasmuch as Schützenberger and Barrière added the sodium hyposulphite to the concentrated solution, and then heated the resulted mixture, I believe the precipitation was due to the above action of heat rather than to a precipitation by the sodium hyposulphite. In a 10 per cent solution of the yttrite group, oxides, I obtained but a slight precipitation with sodium hyposulphite, which, on resolution and treatment, did not again precipitate.

Barrière's methods introduce titanium and zirconium, both of which are exceedingly difficult to separate afterwards.

To obtain the pink colour of the erbium oxide it must be exceedingly pure. One can easily be misled by the colour of these rare oxides when in combination. Some when alone are white, yet when combined are of a dark colour, even brown.

In order to obtain a larger sample of "lucium" to work with, I took 1 kilo. of an average of several hundred samples of North Carolina monazite sand, carefully following Barrière's method, making, however, each separation thoroughly, and failed to obtain any earth answering to the reactions of "lucium," only getting as a result less than 1 per cent of the mixed oxides of the yttrite group of a buff colour and very freely soluble in dilute acids.

Since making these experiments much work has been done by Crookes and others to prove that "lucium" is not entitled to a place in the list of elements, but is impure yttrium.—*Journal of the Franklin Institute*, July, 1897.

BEHAVIOUR OF CHLORAL HYDRATE WITH AMMONIUM SULPHIDE.

By JOSEPH LESINSKY and CHARLES GUNDLICH.

WHILE studying the reactions of chloral hydrate, we observed that, when dissolved in water and mixed with a little ammonium sulphide, it threw down a precipitate only after a certain time had elapsed. This precipitate separated out almost instantaneously after a certain number of seconds had been counted. Upon further experiment we found that, if the concentrations are the same, the precipitate will appear, every time the experiment is tried, in exactly the same number of seconds, provided the temperature is the same.

At first we had considerable difficulty in repeating the experiment, as we had prepared a fresh quantity of ammonium sulphide. It was found, however, that this did not react with the chloral hydrate, even after standing a long time. We were more successful on using a well-saturated solution of ammonium sulphide, and especially a solution that had been standing for several days,—in other words, a solution of the polysulphides.

To show this reaction, the following conditions were observed:—

Two grms. of chloral hydrate were dissolved in 25 c.c. of water, 10 c.c. of this solution run out of a burette into

a narrow beaker, and 5 c.c. of yellow ammonium sulphide poured in, not run in. The liquids must be mixed as quickly as possible. It is best to shake the mixture slightly, and allow it to stand on a piece of white paper, so that the sudden appearance of the precipitate can be seen from a distance in showing the experiment.

At different temperatures the reaction is retarded or hastened, the time necessary for the precipitate to form being inversely proportional to the temperature and concentration.

At 1° C. the separation took place in 44 seconds.

" 20° C.	"	"	19	"
" 45° C.	"	"	8	"
" 65° C.	"	"	3	"

For every 20° rise we have a gradual decrease in time.

The difference between 45° and 65° being 5 seconds.

"	"	45° and 20°	"	10	"
"	"	20° and 1°	"	20	"

The best way to show this reaction is to start a metronome ticking, mix the two solutions quickly, and, while observing the mixture, count the number of seconds it takes before the precipitate appears. The exact nature of this reaction is not understood, especially as the resulting precipitate is evidently a mixture of sulphur and perhaps some sulphur compound of a mercaptan character, the odour being very characteristic. The precipitate was filtered off, and the solution, which was red, extracted with ether, the resulting oil emitting a very powerful odour, and, when diluted with water, suggesting the odour of walnuts. If the concentrations of the two substances are changed, the resulting precipitate will vary in appearance. In our first experiments the precipitate was of a pinkish colour, and the solution red; on heating a little on the water-bath, the precipitate became yellow and then brown, a remarkable succession of changes in colour being observed. In the above-mentioned experiments the precipitate was yellowish brown in colour, the solution itself being yellow, and on warming the precipitate the colour became almost black.

In order to obtain the pink precipitate it is advisable to use an excess of chloral hydrate, but it requires some experimenting to obtain the conditions favourable for the formation of the precipitate.

Perhaps these reactions may be of value in the examination of chloral hydrate as to its purity. It is our intention to study the same, and also to determine the nature of the precipitate and the cause of the "retarded precipitation."

There are only two such cases of retarded precipitation known to us, besides the one described by us, and, even in those cases, it has been impossible to ascertain the causes, although the reactions are not complicated, as in the case with the one described by us.

The reactions are as follows:—

1. The action of sodium thiosulphate upon hydrochloric acid:—



Here the sulphur separates out suddenly after a lapse of time.

2. The action of hydriodic acid with a starch solution when sulphuric acid is added:—



The last reaction is the most characteristic of the three, the iodine separating out so suddenly that most accurate results can be obtained in measuring the time. We have furthermore succeeded in procuring the same results by employing butyl chloral (croton chloral) instead of chloral hydrate, the resulting precipitate being of a beautiful lemon-yellow colour. This last reaction is not as striking

as the others, the precipitate requiring two or three seconds for complete separation.—*American Chemical Journal*, vol. xix., No. 7.

ACTION OF PHOSPHORUS PENTACHLORIDE ON ANILINE AND ITS SALTS.*

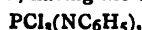
By J. ELLIOTT GILPIN.

THE constant occurrence of chlorphosphuret of nitrogen (PNCl_2)₃ as a by-product in the formation of orthosulphobenzoic acid from commercial saccharine directed attention in this laboratory (Chem. Labor., Johns Hopkins University) to this very interesting substance. Discovered by Liebig, it was studied by Liebig and Wöhler (*Ann. Chem.*, Liebig, xi., 139), Gladstone and Holmes (*Ann. Chem.*, Liebig, lxxvi., 74), Laurent, (*Comptes Rendus*, 1850, Sept. 9), Wichelhaus (*Ber. d. Chem. Ges.*, iii., 163), Hofmann (*Ber. d. Chem. Ges.*, xvi., 1910), and Stokes (*Amer. Chem. Journ.*, xvii., 275). Hofmann studied its action with aniline and paratoluidine, and obtained derivatives in which the chlorine had been replaced by residues of aniline and paratoluidine, the product with the aniline having the composition $\text{P}_3\text{N}_3(\text{NHC}_6\text{H}_5)_6$. Besson (*Compt. Rendus*, cxiv., 1264) has repeated the work done on this substance and obtained the compound PNCl_2 . He says the compound described by Gladstone as having the composition $(\text{PNCl}_2)_3$ and the melting-point 210° is probably a polymeric form of PNCl_2 ; but there is evidently some mistake here, as Gladstone found the melting-point to be 110° and not 210°, while according to Besson it is 114°. As the formation of the chlorphosphuret of nitrogen is due to the action of phosphorus pentachloride on ammonium chloride, the possibility was suggested of obtaining a derivative of it by using instead of ammonium chloride a substitution-product, aniline hydrochloride. Although the substance obtained in this action is not a derivative of the chlorphosphuret of nitrogen, still a comparison of the two forms an interesting study. They show the tendency of phosphorus and nitrogen to combine sometimes in a very stable form, as in the chlorphosphuret of nitrogen and a substance obtained in this investigation, which will be discussed later. The former can be heated with concentrated acids and alkalis without undergoing change; but the trichlorophosphanil, obtained in this work, is very unstable, decomposing with nearly all reagents, and even slowly when exposed to moisture.

Up to the time when this investigation was begun the study of the action of aniline on the chlorides of phosphorus had not led to satisfactory results, and the action with the trichloride only had been studied. By the action of aniline on phosphorus trichloride, Tait (*Zeitsch. Chem.*, 1865, 648) obtained a compound to which he gave the formula $\text{P}(\text{NH}_2\text{C}_6\text{H}_5\text{Cl})_3$. Jackson and Menke (*Amer. Chem. Journ.*, vi., 89) repeated this work, but were unable to obtain the product described by Tait. They obtained a product which they thought had the composition $\text{PCl}(\text{NHC}_6\text{H}_5)_2$, but were unable to isolate it. By dissolving it in alcohol and precipitating with water they obtained the compound $\text{P}(\text{OH})(\text{NHC}_6\text{H}_5)_2$, which they considered to be its hydroxyl derivative. Weyer ("Dissertation," Bonn, 1891) studied the action of aniline on trichloride of arsenic, and obtained two definite products by the successive substitution of aniline residues for the chlorine atoms. These were represented by the formulae $\text{AsCl}_2(\text{NHC}_6\text{H}_5)$ and $\text{AsCl}(\text{NHC}_6\text{H}_5)_2$. The third step he was unable to take. In the present investigation, in which the action of phosphorus pentachloride on aniline and its salts is under investigation, four definite substances have been isolated. They are formed by the substitution of residues of aniline for one or more chlorine

* *American Chemical Journal*, vol. xix., No. 5.

atoms of the phosphorus pentachloride, one being obtained by the action of phosphorus pentachloride on aniline hydrochloride, and three by the action on aniline. The trichlorphosphanil, having the composition—



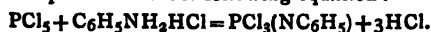
which was obtained from the aniline hydrochloride, shows great similarity to the first derivative obtained by Weyer. They are both very sensitive to moisture, and decompose with water and alcohol in a similar manner. The other substances were more stable, and were obtained from their solutions in alcohol in the form of well-defined crystals.

During the progress of this investigation an article by Michaelis and Schroeter (*Ber. d. Chem. Ges.*, xxvii., 490) has appeared, in which a substance, formed by the action of aniline on phosphorus trichloride, of the composition $\text{C}_6\text{H}_5\text{N}-\text{PCl}_2$, is described. This substance bears the same relation to phosphorus trichloride as the trichlorphosphanil, described in this paper, does to phosphorus pentachloride.

Action of Phosphorus Pentachloride on Aniline Hydrochloride—Trichlorphosphanil, $\text{PCl}_3(\text{NC}_6\text{H}_5)$

After considerable experimenting, the following method was adopted as the best for the preparation of this substance:—Phosphorus pentachloride and perfectly dry aniline hydrochloride are mixed in molecular quantities, not more than 10 grms. of the aniline hydrochloride being used at a time. The intimately mixed compounds are placed in a distilling-bulb, and connected with a condenser. A tube at the lower end of the condenser passes into a vessel of water, to absorb the hydrochloric acid formed and show the rate of the action. The bulb is half immersed in a sulphuric-acid bath. Before the temperature at which the trichlorphosphanil is formed is reached, a small quantity of phosphorus pentachloride sublimes in the upper part of the neck. If the temperature is kept at 170° for six to eight hours, the trichlorphosphanil condenses on the cool part of the bulb, and sometimes in the condenser, as a white coating, covered on the inside with nodules. After the evolution of hydrochloric acid has ceased, the material can be obtained in a very pure condition, if the part in the neck of the bulb is discarded. After being powdered, it is placed in a desiccator over caustic potash to remove the hydrochloric acid. It must be kept perfectly dry, and cannot be purified by crystallisation, as water, alcohol, ether, acetone, and other solvents decompose it. Even when re-sublimed some decomposition takes place. In every case there was a small quantity of a dark, green, sticky material left in the bulb after the action was over. This may have been due to impurities in the phosphorus pentachloride, as the ordinary commercial article was used. When heated with water this green material acts quite violently at first, in consequence no doubt of the presence of a small excess of phosphorus pentachloride, and a tar-like mass is formed, which is unacted on by water, alkali, and dilute acid, and solidifies on cooling. It is soluble in alcohol, with a green colour; so some was dissolved in alcohol and precipitated by addition of water, filtered, and washed. This was repeated several times, and the green powder obtained was analysed; but different preparations varied in composition, and it was evidently not a single definite compound.

The formation of the trichlorphosphanil probably takes place as represented in the following equation:—



Concentrated sulphuric and nitric acids both decompose it, the former with the evolution of hydrochloric acid. Alcohol and ether act on it with the evolution of hydrochloric acid and the generation of considerable heat. Sodium hydroxide acts on it to a slight extent, and changes it to a hard brittle mass. When boiled with water it is decomposed into aniline, hydrochloric and

phosphoric acids, so that the chlorine and phosphorus can be determined in the same specimen, the chlorine as silver chloride, and the phosphorus as magnesium pyrophosphate. To be sure that none of the hydrochloric acid was lost in boiling, several determinations were made by the Carius method, but they agreed with those made in the other way.

The following are the results of the analyses:—

Preparation I.

0.3662 grm. of the substance gave 0.6754 grm. AgCl.
0.2848 grm. of the substance gave 0.5226 grm. AgCl.
0.2560 grm. of the substance gave 0.4672 grm. AgCl.
0.3662 grm. of the substance gave 0.1788 grm. $\text{Mg}_2\text{P}_2\text{O}_7$.
0.2848 grm. of the substance gave 0.1367 grm. $\text{Mg}_2\text{P}_2\text{O}_7$.
0.2560 grm. of the substance gave 0.1242 grm. $\text{Mg}_2\text{P}_2\text{O}_7$.
0.2755 grm. of the substance gave 0.0609 grm. H_2O and 0.3196 grm. CO_2 .
0.4078 grm. of the substance gave 22 c.c. N at 16° and 772.5 m.m.

Preparation II.

0.2568 grm. of the substance gave 0.4635 grm. AgCl.
0.3535 grm. of the substance gave 0.6354 grm. AgCl.
0.3535 grm. of the substance gave 0.1695 grm. $\text{Mg}_2\text{P}_2\text{O}_7$.
0.4237 grm. of the substance gave 23 c.c. N at 11.5° and 762 m.m.
0.3698 grm. of the substance gave 0.0765 grm. H_2O and 0.4081 grm. CO_2 .
0.3646 grm. of the substance gave 0.0748 grm. H_2O and 0.4078 grm. CO_2 .

Calculated for $\text{PCl}_3(\text{NC}_6\text{H}_5)$.	Preparation I.				Found.		Preparation II.		
C	31.55	—	31.63	—	30.09	—	30.50	—	
H	2.19	—	2.45	—	2.29	—	2.27	—	
Cl	46.53	45.59	45.36	45.11	44.43	—	44.61	—	
P	13.58	13.64	13.41	13.56	—	13.40	—		
N	6.17	—	6.38	—	—	6.47	—		

While these results leave much to be desired, they point clearly to the formula given, and are fairly satisfactory considering the instability of the substance.

The sulphate and nitrate of aniline were also treated with phosphorus pentachloride, but they did not act like the hydrochloride. The action of the nitrate was very violent, taking place as soon as they were mixed, without the aid of heat. The whole mass was carbonised in a short time. Paratoluidine hydrochloride was also treated with phosphorus pentachloride, but no volatile product was formed.

Decomposition of Trichlorphosphanil with Water.

When trichlorphosphanil is heated with water it first melts and forms an oil which is decomposed by further boiling. The decomposition gives rise to the formation of aniline, and hydrochloric and phosphoric acids, and can be represented thus:—



The crystals which separated out, after decomposing with water and evaporating, were re-crystallised and analysed. The results of the analyses showed it to be aniline hydrochloride. Another portion was decomposed with water, an excess of barium hydroxide added to liberate the aniline, and this then distilled off. After filtering off the excess of the hydroxide, the presence of barium chloride was proved, showing that free hydrochloric acid had been present. The barium was precipitated with sulphuric acid and the phosphoric acid obtained by evaporating the solution.

Decomposition of Trichlorphosphanil with Sulphuric Acid.

Trichlorphosphanil dissolves in warm concentrated sulphuric acid, with evolution of hydrochloric acid gas. An equal volume of water is added to this, and the liquid is allowed to stand, when a substance crystallised out which

was found to be sulphanilic acid. Several of its salts were made and analysed.

Action of Phosphorus Pentachloride on Aniline. Chlorophosphotetranilide, $\text{PCl}(\text{NHC}_6\text{H}_5)_4$.

When phosphorus pentachloride is slowly added to aniline and the mass constantly stirred, there is immediate action and hydrochloric acid is evolved. The whole mass becomes hot, and after all the aniline has been used up, and further addition of phosphorus pentachloride causes no action, it solidifies. This mass, when cool, is broken up and heated with water to extract any aniline hydrochloride and phosphorus pentachloride present, then filtered, washed, and dried. This product, which is a light yellow powder, is insoluble in water, but slightly soluble in alcohol. It consists largely of one substance, but several others are present in small quantities.

If the dried mass is shaken with two separate quantities of cold alcohol, and the liquid, after being filtered, is allowed to evaporate slowly, besides some needle-shaped crystals there will be found some well-defined monoclinic crystals, which grow to a considerable size. These have to be separated from the other product mechanically. If the solution from which these have been obtained is filtered and allowed to evaporate still further, in some cases, tufts of fine, radiating, silky needles separate out. Both these and the large crystals will be discussed later. After treating several times with cold alcohol, the original material is treated repeatedly with small quantities of hot alcohol, until on evaporation only one substance separates out from the solution. By this process a definite substance is obtained in pure condition. This is slightly soluble in alcohol and insoluble in water. When crystallised from alcohol it usually consists of long irregular-shaped crystals; but when the evaporation is very slow, and absolute alcohol is used, monoclinic crystals, with well-defined basal planes and fundamental prisms, are formed. The composition of this substance is represented by the formula $\text{PCl}(\text{NHC}_6\text{H}_5)_4$, and its mode of formation is expressed as follows:—



It is not decomposed when boiled with water, concentrated alkali, or hydrochloric acid; but when heated with water in a sealed tube to 180° for several hours, aniline, aniline hydrochloride, and phosphoric acid are formed. This substance is characterised by its great insolubility. It is practically insoluble in ethyl ether, acetone, benzene, ligroin, and amyl alcohol, but slightly soluble in ethyl alcohol. When heated, a volatile product, which proved to be a mixture of aniline, aniline hydrochloride, and some diphenylamine hydrochloride, formed by the action of aniline on aniline hydrochloride, was formed, and an amorphous black substance remained. The great stability of this substance is shown by the fact that only a small amount was decomposed when it was heated in a porcelain tube, in a current of oxygen, over the blast-lamp for several hours.

A qualitative analysis of this substance showed that it contains phosphorus, nitrogen, and carbon. The black stable residue is very similar to a product described by Rose (*Ann. der. Phys. Pogg.*, xxviii., 529; *Ann. Chem.*, Liebig, xi., 130). By the action of phosphorus trichloride on ammonia he obtained a product in which he gave the formula $\text{PCl}_3 + 5\text{NH}_3$. When this was heated, ammonium phosphate and ammonium chloride were formed, and a white substance, phospham, PN_2H , was left behind. A comparison of this and the black residue is worthy of notice. Neither is soluble in water or dilute acids. They are only partly decomposed by boiling with fuming nitric acid for some time. Concentrated sulphuric acid attacks the phospham, but acid potassium sulphate is necessary to decompose the black residue. Neither is acted on when heated in a stream of chlorine. They are decomposed by fused alkali, and also with generation of phosphorus or a compound of phosphorus and hydrogen, when heated to

redness in a current of hydrogen. The black residue may also contain a small amount of hydrogen; but this cannot be considered as certain, for the decomposition of the substance is accomplished with so great difficulty that the sources of error introduced may account for the presence of the hydrogen.

The formation of this compound prevented the determination of the carbon and hydrogen by the usual method in the case of all the compounds studied, except that of the trichlorophosphanil, which is volatile. In some cases, when the substances were heated in platinum boats, a transparent film was formed about the small particles of carbon, preventing their complete combustion. The method finally adopted for the determination of carbon, and the only one by which satisfactory results could be obtained, was that described by Fritsch (*Ann. Chem.*, Liebig, ccxciv., 79), in which the substance was oxidised by potassium chromate and sulphuric acid.

The nitrogen was determined both by the absolute and by the Kjeldahl methods. The chlorine and phosphorus were determined as silver chloride and magnesium pyrophosphate respectively, the substance being decomposed by heating it with fuming nitric acid in a sealed tube to 180° for several hours.

(To be continued).

ON THE DOUBLE FLUORIDES OF ZIRCONIUM WITH LITHIUM, SODIUM, AND THALLIUM.

By H. L. WELLS and H. W. FOOTE.

In a previous article (*Amer. Journ. Science*, IV., i., 18) we have described the caesium-zirconium fluorides, and upon comparing these with the corresponding ammonium and potassium salts, which had been previously described by Marignac (*Ann. Chim. Phys.*, ix., 257) it was observed that the types of salts formed varied with the molecular weights of the alkaline fluorides in an interesting manner. The fluorides of smaller molecular weight gave types with a larger relative number of these molecules, while the fluorides of higher molecular weights combined with more zirconium fluoride than the others. This relation is made clear in the following table, which was given in the previous article referred to:—

3 : 1 Type.	2 : 1 Type.	1 : 1 Type.	2 : 3 Type.
$3\text{NH}_4\text{F} \cdot \text{ZrF}_4$	$2\text{NH}_4\text{F} \cdot \text{ZrF}_4$	—	—
$3\text{KF} \cdot \text{ZrF}_4$	$2\text{KF} \cdot \text{ZrF}_4$	$\text{KF} \cdot \text{ZrF}_4$	—
—	$2\text{CsF} \cdot \text{ZrF}_4$	$\text{CsF} \cdot \text{ZrF}_4$	$2\text{CsF} \cdot 3\text{ZrF}_4 \cdot 2\text{H}_2\text{O}$

The present investigation was undertaken with the view, in the first place, of testing the apparent rule with lithium fluoride, which has a lower molecular weight than the fluorides previously experimented upon. Our expectations were realised by the preparation of the salt $4\text{LiF} \cdot \text{ZrF}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$. The salt $2\text{LiF} \cdot \text{ZrF}_4$ was also obtained, but, in spite of a careful search, no intermediate 3 : 1 salt could be discovered. The following table, giving the lithium, potassium, and caesium salts, shows a perfectly symmetrical gradation in types according to the atomic weights of the alkali metals, except that the intermediate lithium salt is missing:—

Type.	Lithium salts.	Potassium salts. (Marignac).	Caesium salts.
4 : 1	$4\text{LiF} \cdot \text{ZrF}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$	—	—
3 : 1	—	$3\text{KF} \cdot \text{ZrF}_4$	—
2 : 1	$2\text{LiF} \cdot \text{ZrF}_4$	$2\text{KF} \cdot \text{ZrF}_4$	$2\text{CsF} \cdot \text{ZrF}_4$
1 : 1	—	$\text{KF} \cdot \text{ZrF}_4 \cdot \text{H}_2\text{O}$	$\text{CsF} \cdot \text{ZrF}_4 \cdot \text{H}_2\text{O}$
2 : 3	—	—	$2\text{CsF} \cdot 3\text{ZrF}_4 \cdot 2\text{H}_2\text{O}$

Marignac's two ammonium salts, 3 : 1 and 2 : 1, also enter the series symmetrically.

We have investigated also the thallous zirconium

fluorides, since the high atomic weight of thallium led us to expect that it would possibly yield a series of salts symmetrical with those of the alkali metals with a still higher ratio of zirconium than was the case with caesium. Such was not the case, however. The salts discovered were:— $3\text{TlF} \cdot \text{ZrF}_4$, $5\text{TlF} \cdot 3\text{ZrF}_4 \cdot \text{H}_2\text{O}$, $\text{TlF} \cdot \text{ZrF}_4$, and $\text{TlF} \cdot \text{ZrF}_4 \cdot \text{H}_2\text{O}$. Two of these three types of thallous salts correspond to types of alkali-metal salts; while one type, the 5:3, is a new one, but the series is not symmetrical with the others according to the atomic weights.

Since Marignac had described but one sodium-zirconium fluoride, $5\text{NaF} \cdot 2\text{ZrF}_4$, and since this differs from all other alkaline zirconium fluorides, we have undertaken a new investigation of the sodium salts. As a result, we have fully confirmed Marignac's results as to the 5:2 salt, which is the one most readily obtained, and we have succeeded in preparing a new salt, $2\text{NaF} \cdot \text{ZrF}_4$, which corresponds to the most usual type of double halogen salts of tetravalent elements. It is evident, however, that the sodium salts, like those of thallium, do not form a symmetrical series with the others.

The following table gives a list of the sodium and thallium salts and shows the positions, "X," of the other compounds prepared by Marignac and ourselves:—

Type.	Lithium salts.	Ammonium salts. (Marignac).	Sodium salts.	Potassium salts. (Marignac).	Cesium salts.	Thallium salts.
4:1	X	—	—	—	—	—
3:1	—	X	—	X	—	$3\text{TlF} \cdot \text{ZrF}_4$
5:2	—	—	$5\text{NaF} \cdot 2\text{ZrF}_4$	—	—	—
2:1	X	X	$2\text{NaF} \cdot \text{ZrF}_4$	X	X	—
5:3	—	—	—	—	—	$5\text{TlF} \cdot 3\text{ZrF}_4 \cdot \text{H}_2\text{O}$
1:1	—	—	—	X	X	$\text{TlF} \cdot \text{ZrF}_4$ $\text{TlF} \cdot \text{ZrF}_4 \cdot \text{H}_2\text{O}$
2:3	—	—	—	—	X	—

While our investigation has shown that the rule for the variation of the types with the atomic weights applies only partially to the zirconium double fluorides, we have shown at least that the variety of types is remarkable, and it is also noticeable that the ratios are nearly the simplest that can exist in such number between the extreme limits 4:1 and 2:3.

Preparation.—Thallium fluoride was prepared by dissolving the metal in sulphuric acid, adding an excess of baryta water, filtering, and passing carbonic acid into the hot solution. The filtrate from this precipitation was evaporated and treated with hydrofluoric acid in excess. The salts were prepared by mixing the acid solutions of the fluorides in varying proportions, evaporating, and cooling to crystallisation. The salts were then removed and pressed between filter papers till dry. In all cases they were stable in the air.

Method of Analysis.—Zirconium and the alkalis were determined by evaporating the salt with sulphuric acid to drive off hydrofluoric acid, precipitating zirconium hydroxide with ammonia and weighing ZrO_2 . The filtrate was evaporated to dryness and the alkali determined as sulphate, either by igniting with ammonium carbonate or heating in a current of air containing ammonia. When thallium was present, the fluoride was dissolved in water, a little sulphurous acid added to make sure that the thallium was all in the univalent condition, and the zirconium precipitated with ammonia. The precipitate needed to be very thoroughly washed. The filtrate was evaporated nearly or quite to dryness to remove free ammonia, diluted to a volume of about 100 c.c., heated to boiling, and potassium iodide added in excess to precipitate thallium iodide. This was collected on a Gooch crucible, washed with 80 per cent alcohol, dried at 100°C ., and weighed.

Fluorine was determined by the ordinary calcium fluoride method after precipitating zirconium with ammonia and removing ammonium salts by evaporation with sodium carbonate. Water was determined by heating the salt in a combustion tube behind a layer of dry sodium carbonate and collecting the water in a calcium chloride tube.

Salts of Lithium.

$2\text{LiF} \cdot \text{ZrF}_4$.—This salt forms when from 0.7 gm. to 2 grms. of lithium fluoride are added to 20 grms. of zirconium fluoride. The crystals are hexagonal, showing prism and pyramid, and rarely a basal plane. In appearance they are very much like crystals of quartz from Herkimer Co., N.Y., but they are very small. On re-crystallising, the 4:1 salt was formed.

Separate crops were analysed with the following results:—

	I.	II.	Calculated for Li_2ZrF_6 .
Li	6.03	6.39	6.42
Zr	41.81	41.64	41.28
F	—	51.62	52.30

$4\text{LiF} \cdot \text{ZrF}_4 \cdot \frac{3}{2}\text{H}_2\text{O}$.—This was the most unsatisfactory salt obtained, though it seems undoubtedly to establish the 4:1 type. As lithium fluoride is very insoluble, only a comparatively small amount could be dissolved in zirconium fluoride, and apparently we could not go far enough toward the lithium end to get the salt in pure condition. It formed in a crust ordinarily, and the crystals were very small. Under the microscope, no mixture with another salt could be found in the crops analysed. Once, however, it was obtained mixed with the 2:1 salt, as seen under the microscope, showing there could probably be no intermediate salt. Various conditions were tried, and crops were obtained from both hot and cold solutions. It forms when 5 to 7 grms. of lithium fluoride are mixed with 20 grms. of zirconium fluoride. On re-crystallising, lithium fluoride is precipitated.

Following are the results of the analyses:—

	Li.	Zr.	H_2O .	F.
I.	9.54	33.14	4.83	—
II.	—	—	4.93	—
III.	9.79	33.30	4.35	53.16
IV.	—	33.23	—	—
V.	—	33.02	—	—
Calculated for $\text{Li}_4\text{ZrF}_{10} \cdot \frac{3}{2}\text{H}_2\text{O}$	9.93	31.91	4.26	53.90

Salts of Sodium.

$2\text{NaF} \cdot \text{ZrF}_4$.—This salt crystallises in very minute crystals of hexagonal outline, coming down in a crust when from one to two parts of sodium fluoride are added to fourteen parts of zirconium fluoride. The salt does not re-crystallise. The following results were obtained from separate crops. The water was probably mechanically included.

	I.	II.	Calculated for Na_2ZrF_6 .
Na	18.66	18.41	18.40
Zr	34.78	36.21	36.00
H_2O	1.96	0.50	—
F (by difference) ..	44.60	44.88	45.60

$5\text{NaF} \cdot 2\text{ZrF}_4$.—Marignac has previously described this salt, which comes down under wide conditions in very good crystals and re-crystallises easily. Prof. L. V. Pirsson has kindly examined the crystals and made the following report:—

"The crystals show good sharp forms, but are very small. They appear distinctly orthorhombic in habit, consisting in the main of rather stout prisms, made up of two prismatic planes, m and m' , and terminated by a rather steep brachydome. In another habit, which is rarer, the front pinacoid, a , is broadly developed, while the prisms are very small; this type also shows at times

a pyramid, β . The plane of the optic axes lies in the base and $a=c$, $b=a$, $c=b$. The optic angle is large, and it could not be told whether a or b was the acute direction. The double refraction is very low. The crystals in their form strongly recall the figures of chrysolite (olivine) shown in the mineralogies."

The analyses gave the following results from different crops:—

	I.	II.	Calculated for $\text{Na}_2\text{Zr}_2\text{F}_{12}$
Na	21'15	21'09	21'23
Zr	33'63	33'55	33'22
F (by difference).	45'22	45'36	45'57

Salts of Thallium.

$\text{TlF} \cdot \text{ZrF}_4 \cdot \text{H}_2\text{O}$ and $\text{TlF} \cdot \text{ZrF}_4$.—These salts crystallise in somewhat concentrated solutions when one part of thallium fluoride is mixed with three or four parts of zirconium fluoride. The analyses invariably show an excess of zirconium fluoride. The hydrous salt crystallises in needles, if the solution be cooled before precipitation occurs. If the solution is evaporated until crystals begin to form and then cooled, the anhydrous salt deposits in minute square plates. The salt gives the 5:3 type on re-crystallising. The following results were obtained:—

	I.	II.	Calculated for $\text{TlZrF}_5 \cdot \text{H}_2\text{O}$
Tl	48'43	47'91	50'05
Zr	22'93	23'16	22'15
F	—	23'17	23'37
H_2O	3'89	4'80	4'43

	I.	II.	Calculated for TlZrF_5
Tl	50'16	49'91	52'37
Zr	23'86	24'08	23'17
F	—	24'32	24'46

$5\text{TlF} \cdot 3\text{ZrF}_4 \cdot \text{H}_2\text{O}$.—This salt crystallises in needles when from 1 to 3½ parts of thallium fluoride are added to 1 part of zirconium fluoride. When about 4 parts of thallium fluoride are added, the same salt crystallises in a different habit, forming prisms of hexagonal outline which under the microscope are seen to be twinned, resembling in this respect the hexagonal-shaped crystals of aragonite. On re-crystallising, both habits give the needle-shaped crystals.

The following analyses were made of the two kinds of crystals. A rather large number of determinations was made on account of the existence of two different forms.

	I.	II.	Tl.	Zr.	H_2O .	F.
I.	61'58	16'88	—	—	—	—
II.	62'05	16'84	—	—	—	—
III.	61'37	17'14	1'40	—	—	—
IV.	61'58	16'88	1'17	19'31	—	—
V.	61'74	17'04	1'42	—	—	—
VI.	—	—	1'31	—	—	—
VII.	62'91	16'42	—	—	—	—
Calculated for $\text{Tl}_3\text{Zr}_3\text{F}_{17} \cdot \text{H}_2\text{O}$	62'47	16'58	1'11	19'84	—	—

$3\text{TlF} \cdot \text{ZrF}_4$.—Crystals of this salt form in brilliant octahedra when one part of zirconium fluoride is added to from four to twenty parts of thallium fluoride. It is easily re-crystallised.

The following analyses were made:—

	I.	II.	Calculated for Tl_3ZrF_7
Tl	72'82	73'20	73'24
Zr	10'91	10'38	10'80
F	15'65	—	15'96

—*American Journal of Science*, iii., No. 18, June, 1897.

NOTICES OF BOOKS.

Journal of the Tokyo Chemical Society ("Tokyo Kwagakukwai"), Vol. xviii., No. 3, March, 1897.

Molecular Conductivity of Amido-sulphonic Acid.—J. Sakurai.—This paper, read before the Society and printed in Japanese characters, takes up about two-fifths of the number, and is illustrated with cuts of the apparatus used. The remaining pages are devoted to Abstracts of Chemical Papers published in other Journals and Miscellaneous Notes. The journal appears to be doing well, but, owing to unavoidable circumstances, we are unable to make any detailed observations on the contents.

Vol. xviii., No. 4, April, 1897.

This number contains an original paper entitled "Report of Experiments on the Manufacture of Japan Camphor (No. II.);" by M. Moriya, Rigakushi. The rest of the number is taken up with the Report on the general Proceedings of the Society, Abstracts of Chemical Papers published in other Journals, and Miscellaneous Notes.

The Agricultural Journal of the Cape of Good Hope. Vol. x., No. 11, May 27, 1897. Cape Town: Townshend, Taylor, and Snashall.

In this number we find an account of some interesting experiments on rinderpest, which is now ravaging South Africa; they were carried out by Dr. Kohlstock under the direction of Dr. Koch. He found that immunisation was produced by serum, but is of short duration, not longer than from ten to twenty days. Serum taken from healthy full-grown animals after mild rinderpest, which had been inoculated with 20 c.c. of virulent blood, without effect, to prove its immunity, gave the strongest serum. Twenty-four cattle were used for this experiment. The best serum was given by an animal which was first injected with gall, and then with rinderpest blood, before immunity was established, so that it suffered from a mild attack: this is the method recommended by Dr. Koch, and has been established by further experiments. The serum seems to be strongest when taken between the tenth and twentieth days—say the fifteenth.

One animal appeared to be naturally immune; it was an ox which had been destined to produce gall; but the introduction of 10, 20, and even 50 c.c. of rinderpest blood failed to produce any effect. Experiments are being made with serum from this animal. Two calves born on the station proved to be immune; they were both produced by cows which had previously been rendered immune. Dr. Kohlstock firmly believes that if gall inoculation is made in the manner indicated, rinderpest will soon be a thing of the past in South Africa: we sincerely hope this is the case.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 25, June 21, 1897.

Examination of some Spectra.—Lecoq de Boisbaudran.—MM. Eder and Valenta resume the critique of my spectrum of Au undertaken by Prof. Krüss. Demarcay has shown that the objections of Prof. Krüss are unfounded. Except the weak ray 433'8, the origin of which I do not presume to assert, but which I always see in the

non-condensed spark with the very concentrated solution of AuCl_3 , M. Demarçay has recognised all my metallic rays. It was, moreover, improbable that the strong ray Au 523.0 could be due to Pd, and that Au 406.4, the strongest of all with a short wire, was alien to gold. The pure gold of commerce would give an appreciable spectrum of palladium, and I did not fail to thoroughly purify my gold. Except 433.8, all my metallic rays have been seen by Eder and Valenta. The other rays (except, possibly, 592.5) are found in the spectrum of the flame of AuCl_3 . The weak ray 592.5 (A.) is difficult to see or to measure. I have obtained it formerly with AuCl_3 and electrodes of Pd or Pt. Having examined it again, I have found it slightly fainter and more cloudy than it is shown in my sketch. Measurements gave about 592.56 (A.). In the flame, the thick band 591.3 would mask 592.5 if this ray exists there. I see therefore always the faint ray 592.5 with AuCl_3 in the spark, but I cannot assert anything as to its origin. Eder and Valenta have a faint and indistinct ray at 592.143 (Rd.), 592.043 (A.). Can this be the same? If I read their text correctly, Eder and Valenta say that my band 560.1 does not belong to gold, no more than my ray 521.2. On referring to my sketches, we find 560.1 and 521.2 among the principal rays or bands volatilised in the flame. Eder and Valenta say that my ray 444.2 does not belong to gold. I have not this ray, but I have one at 443.777 (A.), 443.84 (Rd.), but it has been seen by Eder and Valenta at 443.787 (Rd.). I have not given the ray 434.5, I have only seen there the ray 433.81 (A.), 433.87 (Rd.). I have made new experiments on the subject of this ray 433.8. A series of measurements (difficult) has given 433.83 (A.), 433.89 (Rd.). In a vacuum tube the Cl has a dense cloudy ray, which I have measured at about 434.30 (A.). This seems to me rather remote from 443.83. Moreover, the aspects of the rays are very different. In the open air, between platinum points, the condensed spark gives a thick ray which I find at 434.75 (A.); this is far from 433.83. In the neighbourhood of 433.8 I only see with the condensed spark (taken between dry or moist poles) a faint ray, sensibly more refrangible than that seen with AuCl_3 . The ray AuCl_3 433.8 does not seem to belong to hydrogen, though near to it (Angström gives $H=434.01$). A moderately concentrated solution of AuCl_3 has not shown me 433.8 with the condensed spark. In fine, the ray 433.8 is seen always, though not without difficulty, with the uncondensed spark and a very concentrated solution of AuCl_3 . No more than Demarçay, with his short coil, or Eder and Valenta have I seen this ray between poles of metallic gold, nor by the use of the condensed spark. I do not succeed in identifying it with certainty with any of the rays of Cl, H, C, N, Ar—bodies present in the spark drawn from AuCl_3 .

Potassium Sulphantimonites.—M. Pouget. —Antimony sulphite forms with potassium sulphide a normal sulphantimonite, SbS_3K_3 ; a pyrosulphantimonite, $\text{Sb}_2\text{S}_5\text{K}_4$; a metasulphantimonite, SbS_2K ; and, lastly, a compound containing still less, $\text{Sb}_4\text{S}_7\text{K}_2$.

Fluidity of Melted Nickel.—Jules Garnier.—The great fluidity of nickel when melted at high temperatures may serve to explain the increase of resistance which it gives to irons.

Combinations of Tellurium Iodide and Bromide with the Corresponding Hydracids.—R. Metzner.

Analysis of Bronzes and Brasses by the Electrolytic Process.—A. Holland.—Already inserted.

Formic Aldehyd. Action of Potassa.—M. Delépine.—Not suitable for abstraction.

Destruction of Organic Matters in Toxicology.—A. Villiers.—The author's process is founded on the use of the salts of manganese. Organs such as the liver, the spleen, and the lungs are dissolved in a few minutes. Muscular fibres are disintegrated and then dissolved in about an hour, leaving merely a fatty residue.

Caffeotannic Acid.—P. Cazeneuve and M. Haddon.—Not suitable for abstraction.

Coleopterine: a Red Pigment from the Elytra of certain Coleoptera.—Dr. A. B. Griffiths.—*Pyrochroa coccinea*, *Lina populi*, and *Coccinella septempunctata* yield one and the same pigment, which is soluble in alcohol and ether. The pigment is composed of $\text{C}_7\text{H}_5\text{NO}_5$. When isolated, the pigment is decolourised by light. Its solutions yield no characteristic absorption bands. The author names it provisionally "coleopterine."

"Fracture" of Wines.—M. Lagata.—The author refers this morbid phenomenon to the action of iron.

No. 26, June 28, 1897.

The Death of Prof. Schützenberger.—The President officially informed the Academy of the death of the late illustrious Professor. The deceased, it may be said, died in action, leaving in his laboratory important researches incomplete.

Nomination.—M. de Lapparent was elected a member of the Section of Mineralogy, *vice* the late Prof. des Cloiseaux.

Medical Use of Ozone.—Charles Chardin.—The author recommends a treatment with ozone in cases of cancer and other infectious maladies.

Researches on Nickel Steels, Permanent Magnetic Properties and Deformations.—Ch. Ed. Guillaume.

Silver Sulphantimonites.—M. Pouget.—The action of silver nitrate upon normal sulphantimonite, SbS_3K_3 , yields, according to the conditions of the operation, two well-defined bodies, $\text{SbS}_3\text{Ag}_2\text{K}$, either in an amorphous or a crystalline state. The author has only succeeded in obtaining the salt, SbS_3AgK_2 .

Part of Manganese in certain Oxidations.—Ach. Livache.—In a litharged or manganese oil, the lead or the manganese play the part of intermediaries, taking oxygen from the air and handing it over in continuous manner to the oil, which is thus oxidised more rapidly than it would be in their absence.

The Colour of the Phosphorescence of Strontium Sulphide.—José Rodriguez Mourale.—The experiments prove that the temperature at which the sulphides are formed has not a great influence on the phosphorescence, for the most phosphorescent sulphides are not those whose formation requires the strongest or the most prolonged heat. Strontium sulphide always presents a phosphorescence of a green more or less pure and intense. This property appears closely connected with the properties of production, and with substances which affect the purity of the bodies employed.

Observations on the Molecular Volumes at 0° of Various Crystallised Hydrates of Carbon.—M. Pionchon.

Trioxymethylene and Paraformaldehyd.—M. Delépine.—A thermo-chemical paper, not suitable for abstraction.

On certain Compounds of Phenylhydrazine with the Metallic Iodides.—J. Moitessier.—The compounds here discussed are the phenylhydrazinic zinc iodides, *a* and *b*; the phenylhydrazinic cadmium iodide; the phenylhydrazinic manganous iodide; and the phenylhydrazinic nickel iodide. The nitrates of the various metals of the magnesium series form crystalline compounds with phenylhydrazine.

Compounds of Metallic Salts with the Organic Bases, Homologues of Aniline, and their Isomers.—D. Tombeck.—An examination of compounds formed by toluidine, xylidine, picoline, and lutidine.

Action of Acetylene upon Silver Nitrate.—G. Arthj.—Not suitable for abstraction.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xvii.-xviii., No. 11. June 5, 1897.

Notice on the Life and Works of Alphonse Combes. — Ch. Friedel. — A long and interesting biographical sketch of the career of the late Alphonse Combes, at one time President of this Society. He was a comparatively young man, having been born in 1858, and his death was extremely sudden. The memoir is accompanied by an excellent portrait.

Reaction facilitating the Recognition of Naphthol from Naphthol β . — E. Léger. — An aqueous solution of the matter under examination is made by triturating the crystals in a mortar. To 10 c.c. of this solution are added 2 drops of a solution of hypobromite of soda. With naphthol α a colouration, or even a dirty violet precipitate, is produced; with naphthol β a yellow colour first appears, gradually becoming greenish; it then passes to a brownish green, to again become yellow. It is of importance that all the solutions used should be freshly prepared.

On Two New Alkaloids isolated from a Species of *Jaborandi*. — A. Petit and M. Polonovski. — These two alkaloids have been found in a specimen of the plant for which the name *Pilocarpus spicatus* has been proposed. The alkaloid α is a colourless syrup, with a very alkaline reaction, easily soluble in water, alcohol, and chloroform. The authors have named it *pseudojaborina*; the base β , which they have named *pseudopilocarpine*, presents the same characteristics as pilocarpine, except that it does not act upon polarised light.

Contributions to the Study of Pilocarpine and Pilocarpidine. — A. Petit and M. Polonovski. — These two bodies have been the subject of a good deal of research. At the present time, however, though the properties of pilocarpine are fairly well known, those of pilocarpidine are to a certain extent under a cloud. The authors find the observations of MM. Hardy and Calmels on this subject are not sufficiently exact; this causes them to think that their theoretical conclusions are too arbitrary; they, the authors, have therefore set forth in this note the points of difference they have found, in a somewhat extended research, between their own results and those of the other afore-mentioned experimentalists.

Remarks on the Congealing-point of Milk. — J. Winter. — The author gives as a constant the temperature -0.556° , remarking that variations of 0.01° above or below this can be observed. His results have been confirmed by other workers on the subject.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Chrome Ore. — We understand that chrome ore is used to some extent in chemical works. Can any correspondent oblige with the names of any such works using this ore? — A. & Co.

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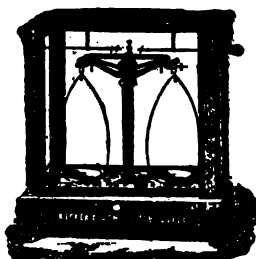
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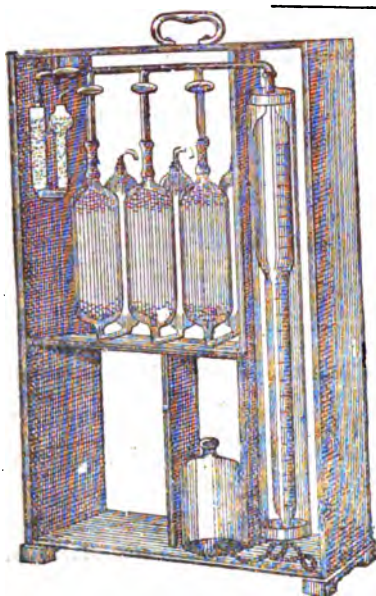
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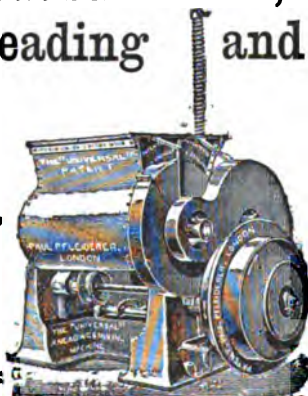
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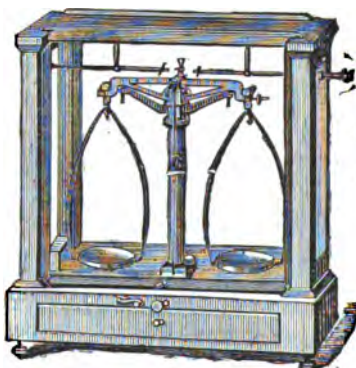


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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1966.

SEPARATIONS WITH ALKALINE ACETATE.

By HARRY BREARLEY.

(Continued from vol. lxxv., p. 254).

II.—Nickel from Iron.

NICKEL is frequently separated from iron by repeated reprecipitation with alkaline acetate. A method previously proposed (CHEMICAL NEWS, vol. lxxiv., p. 16), in which the error introduced by using large amounts of acetate is eliminated by using large amounts of acetic acid with hydrate-free solution, worked very satisfactorily. Further experience with alkaline acetate, under more precise conditions, has shown that accuracy over a wider range may be secured without using so wastefully large a volume of reagents. The present intention is to determine, and state in definite terms, under what conditions the separation can best be made.

It would be too laborious, perhaps unnecessary, to vary the proportion of dissolved hydrate and examine each with series of free acetic acid and acetate. The successful separation in solutions containing no dissolved hydrate which had been formerly experienced led me to work out the series tabulated below:—

TABLE III.

Acetic acid. C.c.	Ammonium acetate. C.c.						Range of temperature turbidity. °C.	
	140.	160.	180.	220.	260.	400.		600.
0	33°0	31°66	30°55	—	—	—	—	86—68
10	—	32°82	31°96	30°4	—	—	—	87—76
20	—	—	32°7	31°77	31°1	—	—	90—82
40	—	—	—	32°47	32°0	30°6	29°89	91—78
60	—	—	—	—	32°68	31°57	30°96	95—83

The work was done on a 5 per cent nickel steel. The figures represent c.c.'s of the potassium cyanide used in titrating the filtrate.

A shorter series was gone through in which the acetic was replaced by hydrochloric or nitric acid, and another in which the sample was dissolved in nitric acid instead of aqua regia. The results obtained were quite as good as those tabulated, but no marked improvement on them. The separations with soda acetate were slightly better than those with ammonium acetate.

Separations from solutions containing the maximum amounts of dissolved hydrate gave—

TABLE IV.

Acetic acid.	Acetate. C.c.		
	5.	10.	20.
0	0°0498	0°0483	0°0477
10	—	0°0500	0°0498
	—	0°0504	0°0504*

The line marked with an asterisk refers to soda salts. The samples actually contained 0°0500 grm. Ni. Experiments were made with solutions containing half the dissolved hydrate. They were neither better nor worse than those already tabulated.

It became necessary to choose what amounts of dissolved hydrate, acid, and acetate should be adopted in order that the effect of certain other variations might be observed. I believe that almost any volume of acetate, with a suitable amount of free acid, could be satisfactorily employed; and, similarly, any proportion of dissolved hydrate might be fixed upon. Chiefly in deference to

customary practice I chose solutions containing the total soluble hydrate.* But such solutions have an irritating tendency to become turbid if they are left for any length of time at this stage; and, moreover, a few c.c. of such dilute acetate as is used made a deal of difference to the separation. For these reasons I add also 10 c.c. of acetic acid. The choice proved a good one.

I will now describe the method for separating nickel and iron. The details rest upon observations either already made or subsequently to be made. It is written as applying to alloys of the two metals; it can, of course, readily be adapted to those elements in other states.

Dissolve in hydrochloric acid and oxidise with nitric acid. In case the alloy contains little or no carbon it may be carbonated alkali until a slight permanent precipitate forms (if the sample is always dissolved in the same amount of acid the volume of standard alkali will be approximately known); add 10 c.c. acetic acid, dilute to somewhat less than a litre (say) with either hot or cold water, add 10 to 12 c.c. ammonia (soda) acetate, of the strength previously mentioned, for each grm. of iron in solution. If, on nearing the boiling-point, owing to imperfect neutralisation, no turbidity occurs, add acetate (2 or 3 c.c. at a time) until it does. Boil, measure volume, cheese until precipitate settles, and filter aliquot part through asbestos. Then cool, neutralise, add measured quantity of dilute ammonia, titrate with KCN and AgI, or estimate Ni in any other way.

The following variations show that the method is widely applicable. They are a selection only from a list embracing every likely variation I could think of.

Dr. Wolcott Gibbs (CHEMICAL NEWS, vol. xi., p. 102; Crookes, "Select Methods," 3rd edition, p. 227), respecting the separation of iron and nickel says:—"The solution from which the iron is to be precipitated must be dilute. Half a litre should not contain more than one grain (0°065 grm.) of the sesquioxide." From a solution containing $\frac{1}{2}$ grm. of iron ($\frac{7}{8}$ grains) to the half litre, I have already separated nickel up to 5 per cent of the alloy. As the separations of nickel from iron by means of soda phosphate in acetic solutions is said to be applicable only when the nickel does not exceed 3 per cent ("Select Methods," p. 205), it was anticipated that with higher percentages the separation with acetate would become less accurate. This fear was not realised, at least not with the somewhat abnormal quantities shown below. Each sample contained 1 grm. of iron.

TABLE V.

Ni taken.	Ni found.	Per cent recovery.
0°1000 grm.*	0°1002	100°2
0°2000	0°2008	100°4
0°3000	0°3009	100°3
0°5000	0°4993	99°86
0°8000*	0°8008	100°1

These were done with ammonia salts. Soda salts were tried in two cases only, and gave—Nickel taken, 0°1000, 0°2000 grm.; Ni found, 0°1002, 0°1996. The potassium cyanide was standardised with quantities of nickel corresponding to that in the filtrates marked with an asterisk. The values for the KCN obtained were not quite proportional. The variation was nearly 1 per cent.

* The "Foreign Science" letter in the CHEMICAL NEWS, vol. xvii., p. 286, chronicles experiments which are interesting in relation to the remarks on dissolved hydrate in my previous paper. It is related that M. Jeannel prepared in the cold a solution of ferric chloride containing *six* times as much iron as the official solution. "A few drops thrown into water produce a voluminous brown precipitate. The solution of ferric chloride is decomposed and precipitated by small quantities of sulphuric acid or of sulphates. It is likewise decomposed by citric, tartaric, or by a few drops of HCl or HNO₃."

† Boiling at this point without the addition of any further reagent forms what is known as Herschel's or Schwarzenberg's method. Dittmar ("Quant. Chem. Anal.," p. 74), in 1887, speaks of it as "the best method." (See also "Fresenius' Quant. Anal.")

The values for the intermediate tests were calculated by interpolation from those found experimentally. No proportions of the two metals less favourable to a perfect separation than the above are likely to be found in practice.

If one has not long been used to perfectly neutralising iron solutions, it will frequently happen, through imperfect neutralisation, that the acetate prescribed is insufficient to even cause a turbidity at boiling-point. In such cases could more acetate be added without introducing error? It has been repeatedly pointed out that the acetate should be added to the cold solution. This precaution probably arose through the almost universal custom of adding wastefully large amounts of acetate. The following experiments answer the question, at least for the present elements and mode of operating.

Two samples containing 1 grm. iron and 0.1 grm. Ni, and in other ways treated as usual, except that no acetate was added, were heated to 90–100° C.; 13 c.c. and 15 c.c. were dropped from a burette, the solution being meanwhile vigorously stirred. Each drop as it touched the iron solution formed a precipitate which was immediately dissolved in the agitated liquid. In the first case a faint turbidity, and in the second case a decided turbidity, was apparent before the last drop of acetate was delivered. There was recovered 0.1001 gr. nickel in each case. The dropping of the acetate and stirring of the liquid seem needless precautions. Two samples, prepared as before, were heated to 97° C., and 15 c.c. acetate were run in and allowed to become mixed by the convective movement of the liquid. The amounts of nickel recovered were 0.1003 and 0.1001 grm. respectively.

When too much acetate has been inadvertently added, and the iron is precipitated at low temperatures, more acid may be added, the assay re-started; or, where neither is desirable, the heating should be discontinued as soon as the precipitate gathers into flocks and the solution against a white rod shows itself only slightly coloured; the flask well shaken, cheesed, &c., as usual. In this way the recovery is greater than if the solution is raised to boiling, although it is not necessarily a complete recovery. On the other hand, if the solution is turbid at boiling-point, but the precipitation of the iron not so complete as to give a clear filtrate, instead of adding more acetate the iron may be further precipitated by prolonged boiling. The table shows that no error is thereby introduced. One grm. iron present in each case.

TABLE VI.

Minutes boiled.	Ni present.	Ni found.
1	0.1000	0.1003
40	0.1000	0.1001
50	0.1000	0.1003

Even when there is no precipitate at boiling-point the separation may be effected by partially confining the steam, and thus raising the temperature. But such an arrangement is troublesome, and has been of no service yet. Under other circumstances it may be.

Additional quantities of ammonium nitrate or chloride act in two ways. They lessen the quantity of alkali necessary to neutralise the iron solution, and they lower the temperature of turbidity. In the accompanying table there was added to I. nothing; to II. ammonium chloride equal to 10 c.c. HCl; and to III. ditto equal to 20 c.c. HCl. The same vol. of acetate, 14 c.c., was added in each case.

TABLE VII.

	Ni added.	Ni found.
I.	0.1000	0.0998
II.	0.1000	0.0998
III.	0.1000	0.0996

Jewett (CHEMICAL NEWS, xl., 273) finds that if ammonium chloride is present the separation of Ni and Fe is more complete than otherwise.

If the nickel is to be estimated by titration with cyanide and silver iodide, it is but a slight disadvantage should the filtrate, owing to imperfect separation of iron, be slightly tinted. Such small amounts may be separated by adding ammonia, or more acetate and boiling and filtering, or by proceeding with the titration as usual. In the latter case, where so much is present as to interfere with the end reaction, a slight excess of the KCN may be added, the solution run through an asbestos filter, and the clear filtrate taken to faint turbidity with the silver nitrate. Comparatively large quantities of suspended ferric hydrate are known to introduce no inaccuracy in cyanide titrations of cupric ammonium solutions, so that examples to justify the practice with nickel are unnecessary.

Obvious enough is the suggestion that in precipitating the iron with alkali and acetate, or alkali alone, adding excess KCN and going back in a filtered aliquot part with silver nitrate, lies a rapid approximate method, such as might be serviceable where a large number of assays had to be completed in little time. Precipitating with ammonium carbonate, adding ammonia, and titrating as just suggested, yielded 99 per cent of the Ni present. The separation was better in cold than in hot solutions, and better when the precipitation was made with carbonate than with ammonia.

There are elements sometimes associated with nickel in the filtrate which are not without action on the cyanide. Of zinc and cobalt, which rarely appear in steel works' laboratories, nothing need be said now. Such quantities of manganese as are found in steel have no influence whatever when ammonia salts are used throughout. But the titrating of a filtrate containing severally 0.05 grm. of nickel and manganese begins to present difficulties when separated from iron as previously described. The ammonia salts present are unable to keep the manganese in solution for any considerable time. In presence of larger quantities of ammonia salts a satisfactory titration may be made with double this quantity of manganese present. I need scarcely point out that if soda salts are used the manganese must be separated at some stage prior to the titration, as suspended oxide of manganese causes low results (CHEMICAL NEWS, i., 25; xxxiii., 152). Copper is conveniently separated from the filtrate as sub-sulphocyanide ("Select Methods," p. 294). Chromium, in the presence of large amounts of acetic acid, would be partly in the filtrate, but with the amount used (10 c.c.) appears to be altogether precipitated with the iron. Large amounts of aluminium would demand exceptional treatment. I propose to postpone this item until the separation of aluminium and iron is considered.

ADDENDA.

Remarks on the Titration.

The means adopted in this paper of estimating the nickel have been largely used and highly spoken of during the past few years. Titration with KCN, in which the indicator was the solubility of the previously formed precipitate in an excess of the reagent, or the decolouration of added cupric ferrocyanide, had been previously used. The use of silver iodide, formed in the solution, as indicator makes the titration as perfect as can be. Denigès (*Comptes Rendus*, cxvii., p. 1078; CHEMICAL NEWS, lxi., p. 42) shows how any compound of silver, or anything capable of modifying such a compound, can be accurately estimated by these means. Moore (CHEMICAL NEWS, lxxii., p. 92) gives a good account of nickel titration after separating that element from steel. The method has also been adopted in the same connection by American writers (Campbell and Andrews, *Journ. Amer. Chem. Soc.*, Feb., 1895).

Before entering on the work previously described, the titration was performed in the presence of varying amounts of reagents. They are not altogether without influence.

The presence of ammonium salts deepens the turbidity formed by the silver nitrate and potassium iodide. If

equal volumes of sulphuric, nitric, and hydrochloric acid are added to separate solutions before neutralising, the iodide precipitate is decreasingly turbid in the same order. Hence, as has been previously recommended, about 2 c.c. of H_2SO_4 should be added unless large quantities of ammonium (or soda) salts are already present.

It is well known that very large quantities of ammonia introduce error into the titration; however, statements on this head are not very precise, and leave much to individual discretion. Denigès says "Variations even very considerable in the proportions of ammonia added, the presence of potash or soda, free or as salts, have no influence on the quantity of silver nitrate used." Campbell and Andrews — "The solution should have just enough free ammonia to give a slight but distinct odour"; and so on. To test the point, ammonia (0.880) was diluted with three times its volume of water. A solution was prepared containing ammonium sulphate, potassium iodide, and ammonia to faint alkalinity, then 4 c.c. in addition, and made up to two litres. 100 c.c. of this mixture was made up to 300 c.c. with water, a measured amount of silver nitrate, and varying amounts of ammonia. The table shows the result with the indicator alone, and ditto to which has been added 0.01 grm. nickel. The two sets of solutions were not identically alkaline to begin with.

Ammon. added	0	$\frac{1}{2}$	2	5	10	20	50 c.c.
C.c. Indicator	5.38,	5.03,	4.93,	4.88,	4.88	4.68,	4.43
KCN (Do. + Ni)	19.27,	19.0,	19.0,	18.95,	18.94,	18.59,	10.6(1)*

* This was several times repeated.

The chloride, nitrate, and acetate of ammonia are without any appreciable influence.

A few drops of artificial alizarin solution may be used as indicator when neutralising the free acid. In an alkaline solution it has a pink colour, and this, along with the creamy silver iodide, produced a purple tint, which changes to pink again when the AgI is dissolved. It is easy to titrate by gas-light in this way.

Moore finds that when the temperature of the solution exceeds $20^\circ C.$, the titrations are irregular. Some laboratories are frequently hotter than this. Solutions which have stood about in our own are at present $25^\circ C.$, but no irregularity has been noticed on this account. Titration certainly ought not to be performed in hot solutions, because else the KCN is decomposed apart from any metal which may be in solution, nor should solutions be unnecessarily exposed to the air, because they absorb CO_2 and give off HCN; hence the slight perceptible odour (Gmelin, vii., 416). But Denigès finds that, in presence of fixed alkali,—"soap-boilers' lye,"—a solution of KCN can be kept unimpaired for an indefinite time, and even boiled for five minutes without appreciable alteration.

I am obliged to Mr. H. Jervis for material, though indirect, assistance.

ON THE VOLUMETRIC DETERMINATION OF ZINC BY POTASSIUM FERROCYANIDE.

By L. L. DE KONINCK and EUG. PROST.

(Concluded from p. 39).

Application of the Method to the Estimation of Zinc in Minerals, &c.

THE titrimetric estimation of zinc by ferrocyanide in acid solution, in minerals and zinciferous metallurgical products, necessitates the preliminary removal of metals susceptible of reacting with the ferrocyanide. The method is approximately the same as that adopted for preparing the solution when using Schaffner's method. To the ammoniacal solution finally obtained, containing a more or less constant quantity of ammonic compounds, are

added a few drops of sodic sulphite; it is then neutralised with hydrochloric acid; then acidulated with a quantity of the same acid, which quantity should be kept constant.

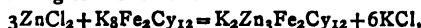
To this solution is added a measured volume of ferrocyanide, constituting an excess of 20 to 25 per cent on the quantity necessary for exact precipitation. After digesting for ten or fifteen minutes, the back titration is performed by means of a neutral or slightly acid titrated solution of $ZnCl_2$. The quantity of zinc corresponds to the excess of the reagent.

Solutions employed.

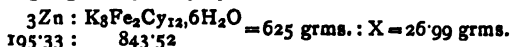
A. A zincic solution $TZn = 0.01$ grm.—that is to say, 10 grms. per litre; it should be slightly acid. This solution is made by dissolving 10 grms. of pure zinc in the smallest possible quantity of hydrochloric acid, in a litre flask, at a moderate temperature. When the solution is complete we bring the volume up to about half the final, neutralise the excess of acid with potassic carbonate, until there is a faint permanent precipitate, which disappears on adding a drop or two of hydrochloric acid. The solution is then cooled to the normal temperature, and the flask filled exactly to the mark with distilled water.

B. A solution of ferrocyanide of potassium. Although in all our experiments we used normal solutions, we think it would be an advantage to give this solution the titration $TZn = 0.00625$ grm., or $\frac{1}{2}$ more than the titration 0.005. When, as is frequently the case in industrial assays, the quantity of zinc present is approximately known, it is best to use 2 c.c. of ferrocyanide per centigram. of zinc expected, and we should then have the excess of about 25 per cent that we recommend.

According to the formula—



the ferrocyanide solution ought to contain per litre the weight given by the proportion—



195.33 : 843.52

This is therefore the weight to take if we have perfectly pure ferrocyanide; if not, we take a little more, and after having found the exact titration of the liquid we dilute it to bring it to the titration wished for. Let us further remark that, on account of the influence of the acid, and of the ammonium chloride, which is generally present in the solutions under examination, it is preferable in every case to determine the titration by direct experiment, and also that it is by no means necessary to know exactly the titration of the liquid, if we do the assays by comparison with pure zinc. In such a case it would suffice to prepare the reagent by dissolving 27 grms. of commercial ferrocyanide weighed on the assay balance.

C. The indicator. A 1 per cent aqueous solution of crystallised nitrate of uranium.

Experimental Determination of the Titration of the Solution of Ferrocyanide, and its Volumetric Relation to the Zincic Solution.

With a view to counterbalancing the influence of acid and salts of ammonia on the estimations, it is important that the determination of this relationship should be made under conditions as similar as possible to those of an assay; we therefore proceed as follows:—

Twenty c.c. of $ZnCl_2$, 100 c.c. of water, 50 c.c. of $AmCl_3$, 2 drops of sulphite,† and 10 c.c. of hydrochloric acid‡ are poured into a flask.

* A solution of $AmCl_3$, containing 200 grms. per litre = 3.75 normal. The proportion of $AmCl_3$ is considerable; it would certainly be preferable to employ less, if possible, but we are obliged to use this quantity in order to reach the proportion which accumulates in an assay by the neutralisation of the ammoniacal solution.

† A solution of 10 grms. of $Na_2SO_3 \cdot 7H_2O$ per 100 c.c. of water. As very little of this is used, and as it changes in contact with the air, not much should be prepared at a time.

‡ Pure HCl, sp. gr. 1.075. This acid is approximately 5 normal and contains about 182 grms. of HCl per litre.

With the exception of the ZnCl_2 solution, which should be measured with an accurate pipette, the measurements may be made with an ordinary graduated glass.

Into this mixture we pour 40 c.c. of ferrocyanide, constituting an excess of 25 per cent; stir well, and let stand ten or fifteen minutes. The precipitate, which was at first gelatinous, has by this time become coherent and settled. We then titrate back with the zinc solution, stirring well after each addition; a tube is more convenient than a rod for the touch test.

After each addition of zinc chloride, a drop of the mixture is taken and let fall into a drop of the indicator. We regulate the quantity of ZnCl_2 to be added each time by the intensity of the colour produced by contact with the uranium salt, and stop when the addition of one or two drops no longer gives a brown colouration, even after two minutes contact with the indicator; this point is shown with great clearness.

The operation we have just described enables us to establish once for all the relation of the value of the two solutions and the titration of the ferrocyanide; in fact, this solution will keep without any change if we keep it away from the light. The slight amount of ferricyanide which may be formed is without influence on the assay if—as we have shown—we add a small quantity of sodic sulphite to the solution to be titrated, as the ferri- is then re-converted into ferrocyanide.

Assay of Minerals.

Preparation of the Solution.—We treat a sample of 2.5 grms. of the mineral, dried at 100° , with nitric acid, in the case of a blende; or with fuming hydrochloric acid, if we have to do with a calamine. After complete solution we evaporate to dryness, so as to make the silica insoluble, and take up the residue with 5 c.c. of fuming hydrochloric acid and a little water; then, after well heating so as to dissolve the basic salts, add 50 or 60 c.c. of water and heat to 70° . We then pass a current of sulphuretted hydrogen; during the passage of the gas we gradually add 100 c.c. of water, to facilitate the precipitation of the lead and cadmium, which would not come down in a too acid solution; but we should not, on the other hand, keep up the current of gas beyond the necessary time, nor dilute the solution too much, or some of the zinc might be precipitated. The precipitated sulphides are collected on a filter, with the silica, if we do not want to estimate the latter separately; and washed with 100 c.c. of water, to which is added 5 c.c. of hydrochloric acid charged with sulphuretted hydrogen.

The washing is complete when the last drops, made alkaline with ammonia, no longer give the slightest precipitate with sodic sulphide; the filtrate is then boiled until all the sulphuretted hydrogen is driven off. We then add 10 c.c. of fuming hydrochloric acid, and 10 to 25 c.c. of saturated bromine water, according to the quantity of iron present, so as to re-oxidise the ferrous salts and to assist the precipitation of the manganese; it is then poured, a little at a time, with constant shaking, into a 500 c.c. matras, containing 100 c.c. of strong ammonia* and 10 c.c. of a 20 to 25 per cent of bicarbonate of ammonia.

On cooling we add water up to the mark, shake well, let the precipitate settle, and filter, but taking care that the filter and all the apparatus is dry.

This method is one which would be used for the most complex minerals. The addition of bromine would not be necessary if there were no manganese present.

We take 100 c.c. of the ammoniacal filtrate, add a few

drops of sulphite, add hydrochloric acid (sp. gr. 1.075) until neutral (about 30 c.c.), then add 10 c.c. more of the same acid.

If after acidulating the liquid becomes brown, owing to the liberation of bromine, it is because the quantity of sulphite is insufficient, and it is then necessary to add a little more than sufficient to cause the disappearance of this brown colour.

Into the solution thus prepared we run such a volume of ferrocyanide, that there may be an excess of 20 to 25 per cent on the quantity necessary for the exact precipitation of the zinc contained in the 100 c.c. of the ammoniacal solution used for the test.

These 100 c.c. contain the zinc from 0.5 grm. of mineral. It is easy to understand, after what we have said previously *à propos* of the value of the titration chosen for the ferrocyanide, that it is necessary to use here a volume represented by the centesimal contents; for example, 38 c.c., if we are dealing with a mineral containing 38 per cent. In practice, we take it as 40 c.c. in round numbers. This has the advantage of permitting the use of calibrated pipettes, which are more easily used than a burette for repeated measurements of the same solution, and also of giving, we think, more accurate results when working quickly.

If we do not know sufficiently nearly the value of the ore, we do a direct assay to determine the volume of the ferrocyanide to be used. For this purpose we take 50 or 100 c.c. of the ammoniacal solution, treat as above, then run in ferrocyanide a little at a time, shaking well, until a test with nitrate of uranium gives, even when tried after waiting some instants, a distinct brown colouration, indicating the presence of a faint, but appreciable, excess of the reagent.

We should use for the final assay a volume higher by one-fifth to one-fourth than that used for the preliminary test; or double this, of course, if we were working on 50 c.c. only. The assay is finished after digesting ten or fifteen minutes, by titrating back to find the excess of ferrocyanide.

By subtracting the weight of the zinc contained in the quantity of the zinc chloride used in titrating back (10 m.grms. per c.c.) from the weight corresponding to the ferrocyanide, we obtain that contained in 0.5 grm. of the mineral; this latter multiplied by 2 gives the centesimal contents in centigrams.

In assays requiring the highest degree of exactitude, such as in cases of dispute, it is preferable to work by comparison; that is to say, to make two parallel estimations—one on the mineral itself, the other on an artificial solution imitating as nearly as possible the value in zinc of the mineral. In other words, on a solution of pure zinc containing as nearly as possible a weight of zinc equal to the weight of zinc in the sample taken of the ore.

This artificial solution is prepared by dissolving in a 500 c.c. flask as much zinc as can be taken up by 20 c.c. of fuming hydrochloric acid, adding to this 100 c.c. of water, then 100 c.c. of strong ammonia and 10 c.c. of the solution of carbonate of ammonium, as with the mineral; after cooling, make the volume up to 500 c.c. with distilled water.

We know that the hydrated precipitates of iron and aluminium produced by ammonia in metallic solutions retain a quantity of zinc by no means negligible (E. Probst and Hassreidter, *Zeit. Anorg. Chem.*, 1892). To obtain complete separation it is necessary to have recourse to double precipitation, which complicates the operation.

By working by comparison we can avoid this complication by adding to the artificial solution, before treating with ammonia, such a volume of ferric chloride of known strength that the weight of iron thus introduced shall be as nearly as possible equal to that contained in the sample of ore taken.*

* Sp. gr. 0.93, or approximately 10 normal. It is desirable to use the smallest possible quantity of both ammonia and of bicarbonate, so as to keep down the contents of ammonium chloride in the final liquid; but it would be well to know if this diminution might not augment the quantity of zinc carried down by the hydrated precipitates of iron and aluminium. Not having had the time to thoroughly study this question, we have adhered to the method generally employed at the present day, for the preparation of the solution for an estimation by Schaffner's method.

* To facilitate calculation, it is best to prepare a solution containing 10 or 25 grms. of iron per litre, and take the necessary volume by means of a graduated pipette.

In this manner the influence of the iron will be neutralised. We operate side by side on the two solutions, taking 100 c.c. of each; acidulating them both with the same quantity of hydrochloric acid, adding the same volume of ferrocyanide, and titrating back with the same solution of zinc chloride after sufficiently digesting the precipitate.

The centesimal value, expressed in centigrams., will be found by the expression—

$$\text{Zn per cent} = 2[0.2 P + 0.01 (N - N')];$$

in which P represents the weight of zinc used for the preparation of the artificial solution, N and N' the number of c.c.'s of the zinc solution at 0.01 of zinc per c.c. used for the back titrations respectively of the 100 c.c. of artificial solution and the 100 c.c. of the solution of the mineral. In fact, as in both cases we have used the same volume of ferrocyanide, the weights of zinc contained finally in each experiment should be equal. For the artificial solution this weight is—

$$\frac{1}{2} P = 0.2 P,$$

augmented by 0.01 grm. for every c.c. of chloride of zinc used; if, for example, it be 0.01 N, then the total will be $0.2 P + 0.01 N$.

For the mineral the weight of zinc will be that contained in 0.5 grm. of the sample; we represent it by X, also augmented by 0.01 grm. for each c.c. of zinc chloride used; this will be $X + 0.01 N'$. From this we get the equation—

$$\begin{aligned} X + 0.01 N' &= 0.2 P + 0.01 N \\ X &= 0.2 P + 0.01 (N - N'). \end{aligned}$$

When working with 50 grms. this must be doubled. It must not be overlooked that if N' be greater than N, the expression $0.01 (N - N')$ becomes negative, and the corresponding quantity must be subtracted from $0.2 P$.

Examples.

I. If the ore contains about 40 per cent zinc, to prepare the artificial solution we take—

$$P = 1.0 \text{ grm.} = (2.5 \times 0.40);$$

then if N = 4.7 c.c. and N' = 4.1 c.c., the zinc present will be—

$$2[0.2 \times 1.0 + 0.01(4.7 - 4.1)] = 2[0.2 + 0.006] = 0.412, \text{ or } 41.2 \text{ per cent.}$$

II. If the ore contains about 30 per cent of zinc, for the artificial solution we take P = 0.75 grm. Then let N = 2.3 c.c. and N' = 3.9 c.c., the zinc present will be—

$$2[0.2 \times 0.75 + 0.01(2.3 - 3.9)] = 2[0.15 - 0.016] = 0.268, \text{ or } 26.8 \text{ per cent.}$$

Direct Titration.

For ordinary daily assays, not requiring such extreme accuracy, we can use direct titration. For this purpose we run the carefully titrated ferrocyanide into the acid zinc solution (warmed to 60° or 70°) until a touch test with nitrate of uranium gives a faint brownish colour. We do not consider the reaction at an end until two touch tests, done at an interval of two or three minutes, both give a colour, and, further, that after the addition of two or three drops of ferrocyanide the touch test should give a distinctly stronger reaction. In direct titration we prefer to use a solution of ferrocyanide, T_{Zn} = 0.01 grm., corresponding to the solution of zinc chloride.

Finally, we give the results obtained by us, by V. Hassreidter, and by Van de Castele, using our process on several ores. In our own assays we did the back titration with a $\frac{1}{2}$ normal solution of chloride of zinc, T = 0.01628 grm.

The ferrocyanide solutions were various and were used in varying quantities; but as all the assays were done by comparison with an artificial solution, and as in every analysis the volume of the two solutions was the same,

it is unnecessary to give the strength and volume of this reagent.

The values given immediately after the description of the mineral are those found by Schaffner's process in an independent estimation by ferrocyanide.

I.

Roasted ore, free from copper, &c.	..	45.28, 45.3% Zn.
Artificial solution	1.125 grms. of pure Zn.	
ZnCl ₂ used in titrating back ..	2.80, 2.92, 2.84, 2.89 c.c.	
Mean	2.86 c.c.	
Ore used	2.5 grms.	
ZnCl ₂ used in titrating back ..	2.73, 2.70, 2.70	
Zn per cent.. .. .	45.42, 45.52, 45.52	

II.

Roasted plumbiferous blende ..	62.86% Zn.	
Artificial solution	1.5 grm. of pure Zn.	
ZnCl ₂ used in titrating back ..	6.02, 6.08, 6.05, 6.04 c.c.	
Mean	6.05 c.c.	
Ore used	2.5 grms.	
ZnCl ₂ used in titrating back ..	5.18, 5.15, 5.08, 5.16	
Zn per cent.. .. .	62.83, 62.93, 63.16, 62.89	

III.

Cupriferous calamine.. .. .	24.30% Zn.	
Artificial solution	0.6 grm. pure Zn.	
ZnCl ₂ used in titrating back ..	11.90, 11.95 c.c.	
Mean	11.925 c.c.	
Ore used	2.5 grms.	
ZnCl ₂ used in titrating back ..	11.90, 11.87, 11.92, 11.95	
Zn per cent	24.10, 24.20, 24.04, 23.94	

IV.

Crude calamine	55.80% Zn.	
Artificial solution	1.375 grm. of pure Zn.	
ZnCl ₂ used in titrating back ..	2.29 c.c.	
Ore used	2.5 grms.	
ZnCl ₂ used in titrating back ..	2.20, 2.18	
Zn per cent	55.30, 55.35	

V.

Calamine No. 1 (Hassreidter) ..	48.3 per cent Zn.	
By the ferrocyanide process ..	48.1 " "	

VI.

Calamine No. 2 (Hassreidter) ..	51.65 " "	
By the ferrocyanide process ..	51.59 " "	

VII.

Blende No. 1 (Hassreidter) ..	36.50 " "	
By the ferrocyanide process ..	36.80 " "	

VIII.

Blende No. 2 (Hassreidter) ..	42.45 " "	
By the ferrocyanide process ..	42.15 " "	

IX.

Van de Castele	38.29 " "	
By the ferrocyanide process (1st assay)	38.34 " "	
	38.27 " "	
" " (2nd assay)	38.41 " "	
	38.39 " "	

The concordance between the different assays of the same mineral shows beyond a doubt that Galletti's method, as we have modified it, merits as much confidence as that of Schaffner. The almost absolute concordance of the results we obtained in the five assays relative to the influence of nitrous acid—minimum, 33.88 c.c.; maximum, 33.94 c.c. ZnCl₂—the last done before we turned to minerals, show the exactitude of which our method is susceptible when used in experienced hands, while the results obtained by MM. Hassreidter and Van de Castele prove that long experience is not necessary in order to obtain satisfactory results. We consider our process to be much simpler and quite as quick as that of Schaffner. We leave it to our confrères, whose position

necessitates them making frequent estimations of zinc, to decide if one of these processes should replace the other, trusting that they will give them an impartial trial.

ACTION OF PHOSPHORUS PENTACHLORIDE ON ANILINE AND ITS SALTS.*

By J. ELLIOTT GILPIN.

(Concluded from p. 44).

Analysis of the Dry Powder.

Preparation I.

THE specimen of aniline used for this was not purified and was very dark, and the products retained some of the impurities.

0.2332 grm. of the substance gave 0.0746 grm. AgCl.
0.19765 grm. of the substance gave 0.0631 grm. AgCl.
0.2102 grm. of the substance gave 0.0553 grm. $Mg_2P_2O_7$.
0.154 grm. of the substance gave 0.3724 grm. CO_2 .
0.1974 grm. of the substance gave 0.4784 grm. CO_2 .

Preparation II.

0.2366 grm. of the substance gave 0.0784 grm. AgCl.
0.2366 grm. of the substance gave 0.0595 grm. $Mg_2P_2O_7$.
0.2096 grm. of the substance gave 0.0691 grm. AgCl.
0.2096 grm. of the substance gave 0.0552 grm. $Mg_2P_2O_7$.
0.488 grm. of the substance gave 0.0633144 grm. N (Kjeldahl).
0.189 grm. of the substance gave 22.23 c.c. N at 25.8° and 758.8 m.m.
0.1584 grm. of the substance gave 0.3863 grm. CO_2 .

Preparation III.

0.1966 grm. of the substance gave 0.0655 grm. AgCl.
0.2025 grm. of the substance gave 0.494 grm. CO_2 .

Analyses of the Crystals.

0.2258 grm. of the substance gave 0.057 grm. $Mg_2P_2O_7$.
0.2309 grm. of the substance gave 0.0792 grm. AgCl.
0.2309 grm. of the substance gave 0.0612 grm. $Mg_2P_2O_7$.
0.2324 grm. of the substance gave 0.0792 grm. AgCl.
0.1613 grm. of the substance gave 0.0553 grm. AgCl.
0.1613 grm. of the substance gave 0.0435 grm. $Mg_2P_2O_7$.
0.1483 grm. of the substance gave 0.0503 grm. AgCl.

	Calculated for $PCl(NHC_6H_5)_4$.	Found (powder).					
		Preparation I.			II.		
Cl	8.14	7.90	7.89	—	8.19	8.14	8.24
P	7.13	—	—	7.44	7.02	7.36	—
N	12.89	—	—	—	12.97	13.04	—
		Found (crystals).					
		Preparation I.			II.		
Cl	8.14	—	8.47	8.42	8.47	8.38	—
P	7.13	7.05	7.40	—	7.53	—	—
N	12.89	—	—	—	—	—	—
C	66.29	65.94	66.09	—	66.50	66.52	—

Attempts made to substitute a residue of aniline for the chlorine atom in the compound, $PCl(NHC_6H_5)_4$, have up to the present been unsuccessful. Some of the powder was added to boiling aniline, in which it readily dissolved; but the product which separated out when it cooled had the same composition as the original material.

Action of Sulphuric Acid on Chlorphostetranilide, $PCl(NHC_6H_5)_4$.

When chlorphostetranilide is treated with concentrated sulphuric acid, hydrochloric acid is given off, and the substance goes into solution. If the acid is warmed and saturated with the tetranilide it becomes solid, with crys-

tals, on cooling. The following method was used for the preparation of the compound formed in the action:—

The tetranilide was treated with as small a quantity of sulphuric acid as possible, and the solution warmed to complete the decomposition and expel the hydrochloric acid. After cooling, water was added, in volume about twice that of the acid, and the solution heated to dissolve the substance precipitated by the water. This solution, on standing, deposits clear octahedral crystals, which can be filtered off, and re-crystallised from water. These crystals are extremely efflorescent, and begin to lose their water of crystallisation before the water which they hold mechanically can be removed.

It has acid properties and is very soluble in water and alcohol, as are also the barium and lead salts, which are formed by neutralising a solution of the acid with barium and lead carbonates, and filtering and evaporating the solutions.

On account of the ease with which these salts dissolve in water, they could not be obtained in a crystallised condition suitable for analysis, but in each case were evaporated to dryness over the water-bath. The action of the sulphuric acid on the tetranilide is as follows:—



The sulphur and phosphorus were determined as barium sulphate and magnesium pyrophosphate respectively. The substance was decomposed according to the Pierson method with nitric acid and potassium chlorate.

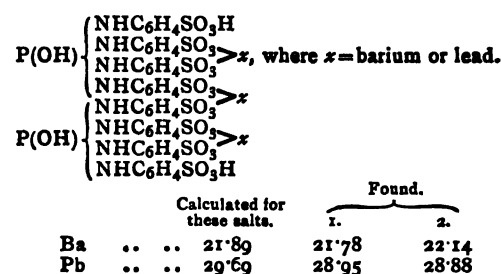
0.1518 grm. of the substance gave 0.1927 grm. $BaSO_4$.
0.2086 grm. of the substance gave 0.0313 grm. $Mg_2P_2O_7$.

		Calculated for $P(OH)(NHC_6H_4SO_3H)_4$.	Found.
S		17.39	17.44
P		4.21	4.19

The determinations of the barium and lead in the salts gave the following results:—

0.4358 grm. of the substance gave 0.1614 grm. $BaSO_4$.
0.4499 grm. of the substance gave 0.1694 grm. $BaSO_4$.
0.6464 grm. of the substance gave 0.2741 grm. $PbSO_4$.
0.6333 grm. of the substance gave 0.2676 grm. $PbSO_4$.

These results cannot be explained on the assumption of the formation of a simple compound; but they agree with a salt of the following composition, in which part of the hydrogen of two molecules of the sulphonic acid is replaced by the metals:—



Action of Phosphorus Pentachloride on the Toluidines.

When the three toluidines are treated under the same conditions as the aniline with phosphorus pentachloride, they give compounds analogous to chlorphostetranilide. The method of purification was the same as that used with the aniline compound. The results of the analyses were as follows:—

Orthotoluidine.

0.2011 grm. of the substance gave 0.0599 grm. AgCl.
0.2888 grm. of the substance gave 0.0692 grm. $Mg_2P_2O_7$.

Metatoluidine.

0.213 grm. of the substance gave 0.066 grm. AgCl.

* American Chemical Journal, vol. xix., No. 5.

Paratoluidins.

0.2918 grm. of the substance gave 0.0814 grm. AgCl.

	Calculated.	Found.		
		Ortho.	Meta.	Para.
Cl ..	7.21	7.36	7.65	6.89
P ..	6.32	6.29	—	—

Besides the compounds already mentioned, two other substances have been obtained by treating aniline with phosphorus pentachloride, under different conditions. The results of the investigation carried out on these substances will be published shortly.

ALUMINUM ALCOHOLATES.

By H. W. HILLYER.

In a former paper (*Amer. Chem. Journ.*, xix., p. 37) we detailed the results of a research in regard to the action of aluminum on ethyl alcohol to which a small amount of fuming stannic chloride had been added. Since sending that paper to the publisher, other observations have been made, which add somewhat to our knowledge of this reaction; and similar studies have been made with methyl alcohol and the two propyl alcohols.

As stated in the article referred to, various anhydrous chlorides, which are soluble in alcohol, were used in an attempt to avoid a difficulty encountered in using mercuric chloride. When absolute alcohol is poured upon chipped aluminum, and either platinic chloride, mercuric chloride, or stannic chloride is then added, a rapid deposition of the metal is soon noticed, and then, with rise in temperature, an increasing evolution of hydrogen followed by a slower evolution, which is long continued, even on application of external heat. A large amount of aluminum is dissolved, enough, in some cases, to make the solution pasty or even solid with the product of the reaction. It has been found that sublimed ferric chloride will also, to a slight degree, bring about the action of aluminum on alcohol, and that even aluminum chloride produces a slight evolution of hydrogen when the chloride is freshly prepared by action of dry hydrochloric acid on metallic aluminum. External application of heat is necessary to the progress of any considerable reaction in the case of ferric chloride or aluminum chloride.

By passing dry hydrochloric acid gas into absolute alcohol standing over chipped aluminum, the metal is readily dissolved with evolution of hydrogen and development of heat. So much aluminum may dissolve in this way that on allowing the solution to cool it becomes solid by separation of a crystalline compound—probably an addition-product of alcohol and aluminum chloride. If, instead of passing into the alcohol a continuous stream of hydrochloric acid gas, a small amount of an alcoholic solution of the gas is added to the alcohol, the action is slower to begin, but increases in vigour till its rate is quite comparable with that induced by the use of anhydrous chlorides of the metals. This form of the reaction is very nicely shown by letting hydrochloric acid gas bubble into the alcohol standing over the chipped aluminum, but removing the gas delivery tube as soon as a fairly rapid evolution of hydrogen is noticed. The reaction will then continue at a good rate, if the mixture is slightly heated occasionally, for more than an hour, until enough aluminum has dissolved to make a jelly when mixed with an equal quantity of water. This hydroxide or basic chloride is, however, entirely soluble in a large quantity of water.

In all the cases noted aluminum chloride has been added, or the conditions were such that it could be formed by decomposition of the hydrochloric acid or anhydrous chloride present. In the case of some of the alcohols it was found, when the stannic chloride was

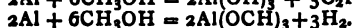
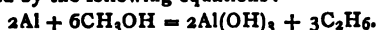
dissolved in a small portion of alcohol, and this solution then added to the main portion of the alcohol, that the action on the aluminum was more vigorous than when the same amount of the chloride was added to the whole of the alcohol. To explain this, it seems to us that in adding the stannic chloride to the smaller quantity of alcohol the temperature was raised high enough to cause the formation of an addition-product of the alcohol and stannic chloride, and that it was upon this compound that the aluminum acted first, and that later it acted on an addition-product of the alcohol with the aluminum chloride which had been found. It is known that all of the anhydrous chlorides used form addition-products with common alcohol. Such addition-products are also known with other alcohols, and it may be said that we obtained a crystalline compound by action of stannic chloride on isopropyl alcohol—probably $\text{SnCl}_4 : 3\text{C}_3\text{H}_7\text{OH}$ as shown by analysis.

For a rapid and satisfactory solution of the aluminum, it may be desirable to have a "couple" of the more easily reducible metals and the aluminum, though our later experiments with hydrochloric acid gas seem to show that it is not necessary.

With the reactions involving the use of stannic chloride and hydrochloric acid gas, it is also necessary to have complete dehydration. When the reaction is in full career, the addition of a little water will nearly or entirely stop the action. In this respect there is another marked contrast with the reactions involving mercuric chloride where the presence of water seems to work no harm.

The observations made would then seem to show that, in general, it is necessary to a satisfactory action of aluminum on alcohol, that it should be anhydrous, that it should contain an anhydrous chloride with which it can form an addition-product, and that the aluminum should be coupled with a more easily reducible metal.

Aluminum Methylate.—The methyl alcohol used was dehydrated by anhydrous copper sulphate, and subsequently distilled. Aluminum acts on methyl alcohol under conditions similar to those favourable to its action on common alcohol. On driving off the excess of alcohol a gelatinous mass remains, but on attempting to distil under diminished pressure the mass does not fuse, but simply crumbles to a black powder and yields no condensable distillate. Only two reactions suggest themselves for the action of aluminum on methyl alcohol. They are expressed by the following equations:—



It was found that the evolved gases, passed through sulphuric acid to retain the alcohol, when mixed with air, exploded violently, and gave no indication of the presence of carbon dioxide when clear lime-water was added to the gaseous residue after the explosion, thus showing the presence of hydrogen only. From this the second equation seems the more probable during the first part of the reaction at least. At a later stage it might be that the nascent hydrogen acted on the methylate previously formed and changed it to the hydroxide, as indicated by the equation—



The following experiments show, however, that this is not the case.

In some cases, if not every case, the aluminum alcohols are soluble in the alcohol from which they are derived. With this thought in mind, an attempt was made to determine whether aluminum methylate had really been formed and decomposed on heating, or aluminum hydroxide had been the final product of the reaction of the aluminum on the alcohol. After a completion of a reaction with aluminum, methyl alcohol, and stannic chloride, a large excess of methyl alcohol was added, and the whole allowed to stand over night. The liquid was decanted from an insoluble residue, and filtered out of

contact with the air. In measured quantities of this solution, the aluminum and the chlorine were determined to ascertain how much of the aluminum could be accounted for as chloride: 10 c.c. of the filtrate were diluted with water, acidified with hydrochloric acid, and the aluminum precipitated by ammonia. From this hydroxide 0.2245 gm. of aluminum oxide were obtained, equivalent to 0.1189 gm. of aluminum. To determine the chlorine, 10 c.c. of the same solution were diluted with water, acidified with nitric acid, and brought to boiling. On adding silver nitrate a precipitate was formed, but it required persistent and very careful filtration to bring the filtrate to final clearness. The reason for this difficulty in clearing, and the cause of the failure of the silver chloride to become curly, has not yet been found. The silver chloride formed weighed 0.4113 gm., equivalent to 0.0897 gm. of chlorine, an amount corresponding to 0.025 gm. of aluminum as chloride.

Considering the similarity of the reaction with this alcohol to those of the other alcohols, and the fact of the evolution of hydrogen during the reaction rather than a hydrocarbon, the presence of aluminum in solution in a form not the chloride can be most easily explained on the supposition that the reaction is normal, that aluminum methylete is formed, but that it is decomposed on attempting to distil it.

Aluminum Propylate.—The normal propyl alcohol used in these experiments was dehydrated by anhydrous copper sulphate. It boils at 97°. In carrying out one of our experiments, 10 grms. of aluminum and 150 c.c. of alcohol were placed in a retort, and then 2 c.c. of fuming stannic chloride were slowly added. No perceptible effect was produced. On applying heat a slight evolution of gas occurred, but ceased on removing the source of heat. On adding 1 c.c. of the chloride an evolution of gas commenced, which was slow at first, but soon became so violent that external cooling was necessary to prevent boiling over. After this spontaneous action became weak, external heating induced further action, which continued several hours until the mass in the retort became quite viscous. In a subsequent experiment approximately the same amounts of aluminum and of alcohol were used, instead of adding the chloride directly to the whole amount of the alcohol, it was first dissolved in a small quantity of the alcohol and then added: 5 c.c. of stannic chloride were dissolved in 10 c.c. propyl alcohol, and of this solution 2½ c.c. were slowly added to the main bulk of the alcohol already standing on the chipped aluminum. Action did not become noticeable for about ten minutes, but then tin began to be deposited; a slow evolution of hydrogen was noticed, which rapidly increased, and soon became violent, as before. The subsequent treatment of the product of reaction was similar to that used with the ethylete. The second of the two operations described above gave a much larger yield of the propylate on distilling, and it showed less indication of the presence of aluminum chloride by fumes at the commencement of the distillation. When the products were re-distilled at a low pressure, they gave an amber-coloured distillate, sometimes solidifying to a clear mass, which gradually became opaque, and sometimes immediately solidified to an opaque solid of the appearance and consistency of fresh commercial grape-sugar. By fractionating, a product was obtained melting at 65° and boiling under 15 m.m. pressure between 235° and 255°. The first fractions were more yellow than the last fraction, which boiled with approximate constancy at 255°. The aluminum in the part boiling at 255° was determined by dissolving in nitric acid, heating the solution till oxidation was no more apparent, evaporating, and igniting the residue. An amount of oxide, Al_2O_3 , was left, which indicated 14.3 per cent of aluminum. By dissolving in hydrochloric acid and precipitating with ammonia, the percentage of aluminum found was 13.2. There should be in aluminum propylate 13.1 per cent of aluminum. The product, made by another operation and distilling between 257° and 262°

at 20 millimetres pressure, was analysed by combustion. In order to protect it from the moisture of the air, it was melted and drawn into weighed glass bulbs with capillary ends, and these, after cleaning and weighing, were dropped into the combustion-tube. The combustion gave—

		Per cent.
H		10.4
C		53.4

The theory requires—

H		10.2
C		52.9

Aluminum Isopropylate.—Isopropyl alcohol is acted on by aluminum in presence of stannic chloride with evolution of hydrogen under conditions like those used for the normal alcohol, but with less apparent readiness. On heating the product under diminished pressure, however, no distillation takes place, but a decomposition like that observed with methyl alcohol.

Amyl alcohol yields a volatile aluminum compound, of a yellow colour, and boiling at 291° C. under a pressure of 12 m.m.

By the use of aluminum as a reducing-agent, and stannic chloride, platinic chloride, or mercuric chloride as the sensitising agent, there seems no doubt that reduction may be performed in entire absence of water, when the substance to be reduced can be dissolved in any of the alcohols, in benzene, ether, or any solvent with which alcohol will mix when it is added to evolve hydrogen with the sensitised aluminum. From later experiments it is doubtful whether the presence of the precipitated tin, platinum, or mercury is essential. Commercial aluminum at least, containing slight impurities of carbon and iron, evolves hydrogen quite freely under the conditions indicated above, by starting the action with hydrochloric acid.

I wish to express here my hearty thanks to Mr. O. E. Crooker, and especially to Mr. R. F. Hastreiter, by whom much of the experimental work detailed above has been done in connection with graduating theses in this laboratory.—*American Chemical Journal*, vol. xix., No. 7.

PREPARATION OF TELLURIUM AT SCHEMNITZ.

ACCORDING to a long description recently published by Herr J. Farbaký in the *Zeitschrift für Angewandte Chemie*, the Schemnitz tellurium works, in Hungary, were established in 1891, and at first the metalloid was extracted from the ore by precipitation with zinc; but since 1895 the process of precipitation by means of sulphur dioxide, proposed by Dr. A. Maly, has been exclusively employed. The first stage is to attack the ore by strong sulphuric acid, to which end 350 kilos. of concentrated acid are raised to boiling-point in a cast-iron pot, into which 150 kilos. of Transylvania ore are ladled with continuous stirring. This results in the decomposition of the metallic carbonates, and the solution of lead, copper, and zinc along with the tellurium and a part of the silver, leaving gold and silica in the residue. The action of the acid is assisted by gradual and progressive heat until the mass is of a syrupy consistency, a stage attained usually in about six hours. The mass is next lixiviated with 250 to 300 litres of boiling water containing 10 to 15 per cent of hydrochloric acid, to extract the soluble compounds formed, the acid acting as a precipitant of the dissolved silver and re-dissolving the tellurium hydroxide thrown down during the dilution of the mass. This operation, with continued stirring, lasts for six hours, and on the following day the liquid and solid materials are separated by filtration under pressure, the gold and silver in the residual cake being recovered in the ordinary manner. The filtrate is run into lead-lined wooden precipitating tanks about 1 m. long by 0.50 m. wide, into which a current of gaseous sulphur dioxide

(purchased in a liquid form in strong cylinders) is passed. At the end of twelve hours the green solution turns brown and black flakes of tellurium begin to separate out; and when a sample of the liquor ceases to give a precipitate with a little hydrochloric acid and sodium sulphite, the operation is at an end. The mother-liquor is absorbed by slaked lime and ashes, and roasted, &c., for recovering the other metals present. The six days required for precipitation in the tanks may be abbreviated to one-fourth or even less by increasing the contact between the sulphurous acid and the liquor—by using carboys instead of precipitating tanks.

The product is usually between 72 and 85 per cent pure, the chief impurity being copper, of which about 6 per cent is present, along with about 8 per cent of tellurium oxide. So far it is not apparent how the copper is precipitated, and experiments on this point are in progress; the process, however, compared favourably in this respect with the zinc process, since the crude tellurium obtained by the latter was only about 29 per cent pure, and contained some 13 per cent of lead, 15 per cent of copper, and 12 per cent of antimony. It was hoped by repeated solution, re-precipitation, and washing to obtain a product 94 to 97 per cent pure, but the process, though efficient on a small scale, is unsatisfactory in practice, besides being expensive, so the product is dried carefully at a low temperature to prevent oxidation, and the mass is fused in luted earthenware crucibles and either cast in sticks of 1 to 2 in. in diameter or granulated.—*Engineering and Mining Journal*.

CARBIDE OF CALCIUM.

SINCE the Order in Council of the 26th February, 1897, in virtue of which certain parts of the Petroleum Acts, 1871 to 1881, were applied to carbide of calcium, the question of the expediency of exempting small quantities of this substance from the operation of the Order has occupied the attention of the Home Office, and the Secretary of State having been advised that such exemption might be safely extended to quantities of carbide of calcium not exceeding 5 lbs., when kept in separate substantial hermetically closed metal vessels containing not more than 1 lb. each, an Order in Council was made on the 7th July, 1897, authorising the keeping of not more than 5 lbs. of carbide of calcium, in vessels as above described, without a license; and the original Order of the 26th February has been amended accordingly. The amending Order appeared in the *London Gazette* of the 9th July, 1897.

It is to be observed that where the carbide of calcium is not kept in vessels as above described, no quantity may be kept without a license.

Whitehall, 23rd July, 1897.

PROCEEDINGS OF SOCIETIES.

SOCIÉTÉ D'ENCOURAGEMENT POUR L'INDUSTRIE NATIONALE.

July 9, 1897.

M. MASCART, President, in the chair.

THE President announced the sad death of M. Schützenberger, Member of the Committee of Chemical Arts. The obituary eulogium, written by M. Troost, was read by M. Aimé Girard.

Letters of thanks were announced from the recipients of the medals and prizes bestowed at the last meeting, and several new candidates for membership were nominated.

CHEMICAL AND METALLURGICAL SOCIETY, JOHANNESBURG.

Annual Meeting, June 19, 1897.

Mr. W. R. FELDTMANN presided.

THE Secretary read the Annual Report of the Council. For the year under review the Randt has been passing through an industrial crisis, which has now reached an acute stage. During the past year several members of the council have resigned, and others have been elected to fill their places. Twelve ordinary general meetings have been held, at which papers have been read and discussed. The membership of the Society has been increased by twenty during the year, and now numbers 107. The financial position of the Society is satisfactory.

The President then gave his valedictory Address, in which he pointed out the great success which had attended the formation and growth of the Society since its inception three years ago, in spite of wars or rumours of wars and commercial depression, and he foretold a respected and prosperous old age.

Mr. BUTTERS was then unanimously elected President and Dr. Loevy Vice-President for the ensuing year.

Mr. E. H. JOHNSON then read some "Notes on the Reduction of Zinc-Gold Slimes," in which he describes an improved method of cleaning up, by using an improvised filter-pump, and well washing the slimes with a 10 per cent solution of sulphuric acid to remove the zinc. He finds one pound of dilute acid to one pound of moist slimes gives very good results. Working with dilute acid, and not heating, he was able to get a perfect settlement within an hour after leaving off stirring. The advantages of this method over the old are—the elimination of the zinc without calcination; immunity from "dusting," the slimes being wet throughout except in the final operation; and the saving of time, which need not be more than three or three and a half days from the commencement of the clean up to having the bars of gold in the safe.

NOTICES OF BOOKS.

Reports on the Experiments on the Manuring of Oats, Hay, Turnips, and Potatoes. Glasgow and West of Scotland Technical College. Glasgow, 1897.

EACH experiment reported on has been conducted on a number of farms, and the conclusions are, as a rule, based on the average results: there are a number of conditions and circumstances which cause great differences in various soils, quite apart from their original composition, such as the effect of drainage, underground water, previous ploughings, manurings, &c. On this account some exception has been taken to average results, on the grounds that they are from a soil which does not exist, but, on the other hand, it may be said that a soil which possesses every property does not exist, so that perfectly accurate results cannot be obtained for comparison.

Averages may be misleading; if, for example, ten farms give one result and ten farms give another, the average of the twenty is practically valueless; but at the same time, over a large number of cases and apart from great extremes, we think the average becomes more and more the standard as time goes on.

The experiments on the manuring of oats were carried out on fifteen farms. The object aimed at was to find if it was profitable to apply artificial manure to oats, and it was found, amongst other things, that superphosphate increases the proportion of grain; that farm-yard manure largely increases the crop, but principally in straw; that readily available nitrogenous manures, such as nitrate of soda and sulphate of ammonia, give considerable and profitable increases.

Experiments on the manuring of hay show that neither bone-meal nor slag are much good the first year,—with chloride of potassium added it is beneficial to clover, more so than with superphosphate; nitrate of soda alone gives large increase of grass, but diminishes the clover; a combination of nitrogenous, phosphatic, and potassic manures is the best, and benefits both grass and clover alike, and gives a highly profitable return.

Some experiments on the use of seaweed as a manure for potatoes shows that seaweed gives a crop quite equal in weight to that obtained with an equal weight of dung; at the same time, however, the "seaweed" potatoes, both in the raw and cooked conditions, were not considered to be of so good a quality as those from the dunged plots.

The last experiments on the manuring of turnips showed that in all soils, in the south and west of Scotland, basic slag, however used, was not so effective in producing an increase in the turnip crop as was superphosphate containing the same amount of phosphoric acid. In peaty or mossy soils different results were obtained; slag is then better when sown with drills than when scattered broadcast; phosphatic and potassic manures without nitrogen cannot be relied on, but a combination of the three makes a good turnip manure. These conclusions are based solely on the amount of yield shown by the weight of the crop.

The Prospector's Handbook. A Guide for the Prospector and Traveller in search of Metal-bearing or other Valuable Minerals. By J. W. ANDERSON, M.A. (Cantab.), F.R.G.S. Seventh Edition. Revised and much Enlarged. Pp. 176. London: Crosby Lockwood and Son. 1897.

SINCE the first edition was published, in 1885, several important discoveries and openings up of metal-producing countries have occurred. South Africa and Western Australia are the most important of these, though we are now hearing remarkable stories of the fabulous wealth lying about in the Klondike and Yukon regions of North-West Canada.

The geological lessons learnt in South Africa are of great importance, in that they teach us to prospect with eyes and mind open to new impressions and ideas, instead of only looking out for such indications which old experience has taught us to associate with gold-bearing strata.

Among the additions to the Handbook may be mentioned the reference to aluminium ores. This metal, as is well known, is not found in the free state, but generally in combination with silica, oxygen, and fluorine. Its principal ores, besides corundum, are beauxite and cryolite.

The general arrangement of the book is the same as in the previous editions; it comprises chapters on prospecting; blowpipe testing; the character, description, and occurrence of rocks, minerals, and metallic ores; the assaying and treatment of ores; and lastly surveying. There is an Appendix containing the usual tables we expect to find, and very useful they are sometimes, when miles away from everywhere; and a Glossary of terms used in connection with prospecting, mining, &c. Young prospectors should beware of the Glossary, lest, by learning too much of it, they "give themselves away," and show their inexperience.

Reports of the Government Analyst for British Guiana for 1894-5, 1895-6, and 1896-7. Georgetown, Demerara: C. K. Jardine, Printer to the Government, 1895-6-7.

THE work done in the Government Laboratory has increased from 2097 samples in 1894-5 to 2399 samples in 1896-7, and we note that the number of official samples has decreased in that period from 1472 to 1450; but in the intermediate year, 1895-6, there was a large increase

of Government work, viz., 1547 samples. As is to be expected, by far the largest number of samples examined were of sugar-cane, sugars, and articles of food and drink. It is remarkable to observe that during these three years no fewer than 38, 46, and 12 samples respectively were of human viscera, in cases of suspected poisoning. We thought a knife or machete was the favourite instrument for argument in the West Indies. But we are glad to see that the latter year, 1896-7, is the lowest (with 1893 when it was the same) in such cases of suspected poisoning ever recorded in the colony.

Report of the Agricultural Work in the Botanic Gardens for the Years 1893-4-5. British Guiana. Georgetown, Demerara: C. K. Jardine, Printer to the Government. 1897.

THE Report over the past three years has been delayed, with the object of recording the termination of the period of the first set of manurial experiments with sugar-cane. This period came to an end in December, 1895, and, as many experiments overlapped, it was considered undesirable to report at arbitrary intermediate periods.

The first experiments discussed in this Report are those with seedling canes, and, as has been pointed out in a previous Report, it is found to be impossible to form any opinion of the richness of the seedling progeny from the richness of the actual parent, on account of the range of variation being so great, and we are forced to regard the saccharine richness of a seedling equally problematical, as are conjectures beforehand as to its colour and size. It is believed that, in the future, improvement lies in raising year by year new varieties from seed, selecting those of greatest vigour, most abundant field yield, and high saccharine contents, in the hope of obtaining a new variety: the "pedigree" seedlings already obtained by this means show that the experimenters are on the threshold of this advance.

The general deductions as to the action of manures and lime upon four crops of sugar-canes grown upon very heavy clay land may be briefly summarised thus:—

1. That nitrogen in the forms of sulphate of ammonia, nitrate of soda, and dried blood, is without doubt the manurial constituent the supply of which mainly governs the yield of the plant.

2. When applied in quantities capable of supplying not more than 40 lbs. of nitrogen per acre, there was practically no difference in the effects of sulphate of ammonia and nitrate of soda, but the former was cheaper. Dried blood was decidedly inferior. The best results were with one-third nitrate of soda and two-thirds sulphate of ammonia.

3. From 2½ to 3 cwt. of sulphate of ammonia per acre appeared to be the most certainly profitable application of nitrogen.

4. Upon plant canes, superphosphate of lime gave considerable and profitable increase of yield when added to manurings of nitrogen and potash.

5. Mineral phosphates require to be applied in such heavy dressings as to render their use unprofitable.

6. Slag phosphate appears to be a promising source of phosphate for plant canes, instead of superphosphate of lime.

7. The addition of potash has little effect; nitrate of potash instead of nitrate of soda was unsatisfactory.

8. The use of lime resulted in largely increased yields.

It is satisfactory to note that the West Indian sugar colonies have not been tamely allowing themselves to be driven out of the sugar market, and the colonies themselves destroyed, by the hostile German Sugar Bounties. The planters and others of the colony of British Guiana, we are informed, make great use of the scientific facilities placed at their disposal by the Government; and we are glad to be able to correct the idea, which is perhaps too widely prevalent, that the tropical colonists of Great Britain take but little interest in the scientific aspect of

their work. Reports such as the one now before us show conclusively that the present-day planters are not content to follow the old methods of a century ago, but are doing their best to help themselves in their almost hopeless struggle against the effects of German Sugar Bounties.

CORRESPONDENCE.

PRECIPITATION OF COPPER BY MAGNESIUM.

To the Editor of the Chemical News.

SIR,—In connection with the subject of the displacement of copper by magnesium in solutions of copper sulphate, the following note may be of some interest.

The action appears to be conditioned by the purity of the copper sulphate.

Specimens of bright magnesium ribbon were immersed in an excess of solution—(1) of ordinary blue vitriol, (2) of re-crystallised copper sulphate, bought as pure, and (3) of a specimen of copper sulphate which had been fractionally re-crystallised by myself four times from 2 kilos. of a sample equal in initial purity to (2).

After immersion for two and a half hours, the second specimen was found to have deposited a very small trace of copper. In the third instance the magnesium was withdrawn apparently as bright as when first immersed. On solution it was found to contain a minute trace of copper.

The action appears to be accelerated as time goes on, but even after four days a considerable amount of magnesium is found in the solid residue.

On the other hand, the purest zinc at my disposal was instantly covered with a deposit of copper in each instance, and the action was found to be completed in twelve hours, no trace of zinc being discoverable in the washed pulverulent copper.

I regret that other scientific work has prevented me from continuing these experiments, but I hope that in the course of the winter one or two of my students will have something to say on the action of purified magnesium on purified copper sulphate solutions.—I am, &c.,

SIDNEY A. SWORN.

Chemical Laboratory,
Municipal Technical School,
Gravesend, July 27, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Bulletin de la Société Chimique de Paris.

Series 3, Vol. xvii.-xviii., No. 12, June 20, 1897.

M. Freundler has studied the decomposition of pyromucates of the alkaline earths under various conditions. This decomposition gives rise to the formation of a small quantity of furfuran, to a carbide, C_3H_4 , and to oxide of carbon.

M. Tardy has separated carbides from bitter fennel and found many interesting bodies.

M. Ponsot, in thinking over a cryoscopic experiment from the calorimetric point of view, finds a means of detecting whether a cryoscopic method regularly used has systematic errors.

On Yellow Light for Polarimetry.—F. Dupont.—The author finds that a mixture of common salt and tribasic phosphate of soda, fused together in proportions similar to their molecular weights, answers perfectly and gives excellent results. The mixture melts more easily

than sea salt, does not decrepitate, and gives a remarkably steady yellow light. Polarimetric observations are by its use very easy and exact.

Precipitation of Chloride of Copper by Aluminium.—J. B. Senderens.—A reply to the remarks of M. Tommasi.

On Borate of Lithium.—H. Le Chatelier.—Lithium shows several analogies, both to the alkaline metals and the alkaline earths. The author has endeavoured to find whether its borates, even by their composition, show more precise analogies, considering the great variations observed between one metal and another, in the number and composition of the borates of each.

Basic Salts of Cadmium.—M. Tassilly.—Already inserted.

On Dextro-Licarhodol.—Ph. Barbier and G. Léser.—Pure licareol is heated for eight hours at 150° or 160° with its own weight of acetic anhydride. At the end of this time the acetic acid is removed by successive washings, the product of the reaction is dried and rectified *in vacuo* in three fractions: the first, from 50° to 80° , consists principally of a mixture of tetratomic terpenes, $C_{10}H_{16}$; the second, from 80° to 105° , which still contains some unacted upon licareol, is again submitted to the action of acetic anhydride, but under modified conditions; the third, from 105° to 130° , submitted to a minute fractionation *in vacuo*, gives an abundant colourless liquid, of agreeable odour, boiling at 119° to 120° under a pressure of 10 m.m., and which has the composition of the acetic ether of an alcohol, $C_{10}H_{18}O$.

On some Properties of Caffeine.—E. Tassilly.—Crystals of caffeine from aqueous solution were carefully dried for a week; they then gave on analysis the formula $C_8H_{10}N_4O_2 \cdot H_2O$. On heating to 50° — 55° this body commences to lose weight, and on increasing the temperature this loss of weight continues; but the whole of the water of crystallisation is not driven off even at 150° , while the caffeine itself is volatile at this temperature. Experiments showed the caffeine is carried off by water vapour when evaporated on a water-bath, or even when heated up to 110° . Further experiments were made on the action of alkalis on caffeine, and it was found that though magnesia has no action, the same cannot be said of baryta and lime, which decompose it with the production of ammonia.

On an Isomer of Disulphide of Diphenylene.—P. Grenesse.—This body is formed while preparing disulphide of diphenylene by the action of sulphur on benzene, in the presence of chloride of aluminium; it is almost insoluble in all solvents, and takes an emerald-green colour under the influence of sulphuric acid.

Decomposition of Pyromucates of the Alkaline Earths by Heat.—P. Freundler.—This paper deals with furfuran and its preparation; the only practical method of preparing this body is that of Limpricht, who obtained it by the distillation of pyromucate of barium with soda-lime. *In vacuo* the decomposition of pyromucates takes place at a lower temperature, but a small quantity of acetone is also formed; under pressure pyromucates can be heated up to 375° without undergoing any change, but towards 400° it decomposes brusquely.

On the Carbide C_3H_4 , a Secondary Product of the Decomposition of Pyromucate of Barium.—P. Freundler.—This carbide fixes bromine in the cold and gives a small quantity of a dibromide boiling at about 50° *in vacuo*; it is extremely irritating to the eyes. The principal product, however, is a liquid tetrabromide, $C_3H_4Br_4$, boiling at 162° under a pressure of 20 m.m. This latter body has not been obtained quite pure, on account of its hygroscopicity and because it loses hydrobromic gas rapidly at ordinary temperatures.

On some Derivatives of Piperonal.—S. Baude and A. Reyckler.—Piperonal easily lends itself to the preparation of a series of derivatives similar to those prepared from anisaldehyd, such as methylenedioxybenzylate of

ethyl, methylenedioxyphenylpropionic acid, and methylenedioxyphenylacetylene.

On Amidised Amidines.—Charles Lauth.—The author has prepared two amidines giving similar colours, but they are not very stable under the influence of light.

Intervention of Manganese in the Oxidations brought about by Laccase.—G. Bertrand.—Some time ago the author found, while studying the chemical composition of laccase, that the ash contained a relatively high proportion of manganese, but he did not then estimate it. He has now returned to the subject, and has found as much as 2.5 per cent of manganese present. In the action of the ferment he is of the opinion that manganese cannot be replaced by any other metal, not even by iron.

Estimation of the Oxygen Dissolved in Sea-water.—A. Levy and Felix Marboutin.—Already inserted.

Estimation of Cream of Tartar in Wines.—H. Jay.—Several writers on the subject of the estimation of bitartrate of potash rely more on methods of crystallisation than on precipitation; they assume, in fact, that the former is more exact. The author has carefully investigated the subject, and arrives at the conclusion that the results, intrinsically and directly inexact, obtained by the methods of Berthelot and of Fleurieu, are by their corrections, which are constant, nearer the truth than those obtained by crystallisation, in which he finds the excess is not constant.

On the Yellow Colouring-matter of the Oil in White Wines containing Caramel.—Alberto D'Aguiar and Wenceslau da Silva.—Already inserted.

Commercial Rectification of Organic Products.—E. Barillot.—An interesting paper, which, however, requires the accompanying diagram.

MISCELLANEOUS.

Seventy-seventh Annual Announcement of the Philadelphia College of Pharmacy, 1897.—Previous to 1840 pharmacists were not recognised in pharmacopoeial conventions, but in that year the College was invited to co-operate with the Committee on Final Publication and Revision. Since then pharmacists have so improved their position that at the last convention in 1890 they numbered sixteen of the twenty-six members. Since the establishment of the institution 14,661 students have matriculated, and 4416 persons have taken the degree of Graduate in Pharmacy.

Paris International Fire Prevention Congress, 1897.—The terrible catastrophe at the Charity Bazaar in May last has determined a number of influential men to form a committee, and call together an International Congress, to discuss all means to prevent and minimise fire risks in theatres, concert halls, and places of public resort. The date of the congress has not yet been finally decided on, but will be announced shortly. In conjunction with the Congress there is being organised an International Exhibition of all engines, inventions, products, and plans for the prevention and extinguishing of fires. Inventors, manufacturers, and engineers are invited to exhibit their machines and inventions; in this way scientific discussion will be strengthened by practical demonstration. One hundred and fifty Senators, Deputies, Municipal Councillors, and scientific men have already joined the committee, and the British Secretary, Mr. Frederick Hoare, 249½, High Holborn, will be glad to receive any data or useful information bearing upon the business of the Congress. France is decidedly behindhand in this matter, and we doubt if she can get outside help better than in England.

Wanted, place as Assistant at Chemical Laboratory or Porter, by experienced Young Man, aged 24; single. Good references.—Address, R. Baggott, 9, Shroton Street, Lisson Grove.

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DAVID LEWIS,
Treasurer's Chambers, 20, York Place,
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FORTHCOMING EXAMINATION.

Dispenser in H.M. Naval Hospitals at Home and Abroad (20 to 25), 25th August. The date specified is the latest at which applications can be received. They must be made on Forms to be obtained with particulars from the Secretary, Civil Service Commission, London, S.W.

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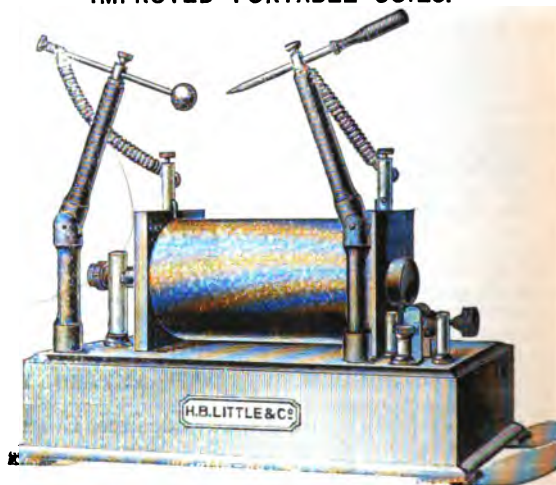
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Vol. LXXVI., No. 1967.

RECENT PROGRESS OF ALCHEMY IN AMERICA.

Reported by H. CARRINGTON BOLTON, Ph.D.

THE suicide of Dr. James Price, the Fellow of the Royal Society who preferred ignominious death to an investigation of his claim to success in transmuting base metals into silver and gold, was a heavy blow to the pretensions of alchemists in England, for we hear but little of the disciples of Hermes in Great Britain during the hundred years that have elapsed since that tragic event. Of vulgar swindlers, such as the American confidence-men who attempted to cheat the Bond-street jeweller by a pretence of "multiplying" sovereigns, there are occasional examples; and there have been instances of successful imposture practised on the Royal Commissioners of Patents, who have granted patents for "getting gold from wheat" by skimming water-washed straw, and for detecting underground treasures by a certain divining-bottle; but these are exceptional, and need not be chronicled in sketching the progress of alchemy in the nineteenth century.

In the United States of America the claims of alchemists have rarely been seriously considered, the people being free from the fetters of authority and tradition, and possessing a shrewd, calculating spirit, symbolised in the word "Yankee." A smart and educated French chemist, named Paraff, visited America a few years ago, pretending to know a process for converting copper into gold, and for a while he succeeded in transferring gold from the pockets of ignorant persons to his own; but when the courts of Peru required him to conduct a transmutation in their presence the career of this modern Cagliostro came to a disastrous end. Within the last twelve months, however, two claims to success in transmutation, or creation of gold, have been made with such boldness, publicity, and persistency, by persons assuming to have superior scientific qualifications, that it has been impossible to ignore their representations, and in one instance the Director of the United States Mint has been called upon to endorse or condemn the secret process of the inventor.

The first of these claims was announced in August, 1896, by Dr. Stephen H. Emmens, a well-known chemist of New York city, whose name is attached to the high explosive "Emmensite" invented by him. Dr. Emmens is a member of several learned societies, and author of papers on chemistry, electricity, and metallurgy, as well as of several novels and poems. In his "Argentaurum Papers" he presents erudite considerations respecting the Newtonian doctrine of gravitation.

To avoid doing injustice to Dr. Emmens the following account of his discovery is given almost wholly in his own words:—

The Transmutation of Silver into Gold.

"Our work, which converts silver into gold, had its origin in the course of certain investigations which I undertook for the purpose of preparing chemically pure nickel. This was in the year 1892. Commodore Folger, who was then Chief of the Bureau of Ordnance of the United States Navy Department, had forwarded to me for investigation a very remarkable specimen of rustless nickel steel which it was proposed to use as a material for torpedo netting. I found the physical properties of this material to be so extraordinary that I desired to investigate the physical behaviour of a similar alloy made with

absolutely pure iron and pure nickel. In attempting to prepare these pure metals, a certain product was obtained which seemed to differ from anything recorded in the textbooks. The same product was subsequently found when the investigation was extended to the case of metallic cobalt. And, finally, those who were associated with me in the investigation agreed with me in considering that the phenomenon observed afforded indications of the existence of some substance common to the whole of the elements in what is known as Series 4 of Group 8 of the Classification of Chemical Elements, now universally adopted by scientists, in accordance with what is known as the 'Periodic Law of the Elements.' We did not further pursue the particular line of investigation upon which we had set out, because it appeared to us almost self-evident that if we were right in supposing a common substance to be present in any single series of elements the same would hold good for each group.

"And as Group I. of the classification contains the precious metals—gold and silver—it was obvious that our time and attention should be directed to these metals rather than to any others. . . .

"It is, of course, out of the question for me to make public the whole of our knowledge in the matter; but I may without danger to our interests give a general explanation of the work which will be satisfactory to the scientific world.

"Our starting-point, so far as silver and gold were concerned, was afforded by the remarkable discoveries of Mr. Carey Lea with regard to the changes that could, by laboratory methods, be induced in the molecular structure of metallic silver. That gentleman discovered a means of causing silver, while still in a metallic condition, to enter into aqueous solution. In other words, he discovered a method of reducing metallic silver to a condition of extremely minute subdivisions. It was found, as might have been expected by anybody familiar with the periodic law of the elements, that this subdivision of metallic silver was attended by very considerable changes in the physical properties of the substance. The inference was obvious that, if such subdivisions could be pushed a stage further, the silver molecules would become dissociated if they were in themselves of composite structure; and, as all chemists have long been agreed respecting the reality of such composite structure, we felt absolutely sure of our ground.

"Accordingly, when by certain physical methods and by the aid of certain apparatus, we succeeded in bringing about a further subdivision of the silver, we were not surprised to find that the substance obtained differed so far from ordinary silver that it could no longer be regarded as the same elementary substance. It seemed to require a new name and a new chemical symbol. Inasmuch, therefore, as our theory was that this substance was common to both gold and silver, and in reality was the raw material out of which both gold and silver were constructed by the hand of Nature, we named the substance "Argentaurum." We gave it also the chemical symbol "Ar."

"The next step was to ascertain whether this substance could be so treated as to be grouped into molecules of greater density than those of silver. Here the element of personal danger was introduced into our researches, and the success of our work on a commercial scale has yet to be assured by the construction and safe manipulation of new apparatus, in which vast energy will be employed. Working upon the necessarily microscopical scale of our experimental researches, we found that the substance called by us 'Argentaurum' can be aggregated into molecules having a density considerably superior to that of silver molecules, and, we think, identical with that of ordinary gold molecules. Whether we are right as to this or not, the condensed argentaurum presents the appearance and is endowed with the properties of ordinary metallic gold. For example, it is green by transmitted light and yellow by reflected light,—properties which, as

all chemists know, are possessed by gold alone. Its resistance to the action of either nitric or hydrochloric acid alone, and its solution by a mixture of these acids, are also distinguishing properties of pure gold, and of no other yellow metal. Under the microscope it is indistinguishable from ordinary gold."

After some comments on the Periodic Law, Dr. Emmens continues:—

"If Mendeleeff's table be examined, it will be seen that a vacant space exists in the Sub-group of Group I., and that this vacant space stands immediately between silver and gold. Our claim is that the hitherto missing element in question is our argentaurum, which in itself therefore is neither silver nor gold, but which may, by our new physical methods, be converted into gold."

Referring to the question of cost the discoverer says:—"We do not consume any chemicals and other costly materials in our process; what we use is mainly energy in some of its various forms, such as heat, electricity, magnetism, gravity, cohesion, chemical affinity, X rays, and the like." "Our chief source of expense is the time required for bringing about the desired molecular changes." And further discussing the expenses, Dr. Emmens estimates that "one ounce of silver will produce three-quarters of an ounce of gold," and that "we can reckon on a profit of at least three dollars per ounce upon all the silver we employ."

In a pamphlet, published in July of this year, Dr. Emmens states that the process of transforming silver into gold depends largely on mechanical treatment, and hints at "the combined effect of impact and a very low temperature." He now operates on Mexican dollars, and, as substantial proof of his labours in transmutation, he has sold to the United States Assay Office six ingots of an alloy of silver and gold, aggregating in value 954 dollars and 80 cents. These ingots have been sold since April 13th, and have been delivered at the rate of two per month; they varied in weight from 7 ounces to 16½ ounces.

Dr. Emmens remarks in this connection, "The gold-producing work in our Argentaurum laboratory is a case of sheer Mammon-seeking; it is not being carried on for the sake of Science, or in a proselytising spirit; no disciples are desired, and no believers are asked for. I have every confidence that the production of Argentaurum gold will be brought up to 50,000 ounces monthly within a year."

The second claimant for a process of converting base metals into precious ones is Edward C. Brice, of Chicago, who has lived a rather chequered life as an enlisted man in the Army, a member of the Police force of Washington city, a promoter of a scheme for manufacturing a patent brick, and a discoverer of the Philosopher's Stone. According to the newspapers, Brice began his operations about three years ago, in Washington, by interesting some rich and credulous men in a secret process for transmutation; several experiments made in their presence seemed to confirm the promoter's statements, but one of those who was supplying money became suspicious, and thought he detected the introduction of gold-leaf into the crucibles used; and later, by skilful diplomacy, he learned from Mrs. Brice that her husband had been buying much gold-leaf for gilding picture-frames! This unkind exposure seems to have caused a temporary suspension of Brice's operations, but in no wise discouraged him, for he next appears in Chicago, where he pushed his old schemes with great success, securing the financial backing of some of the prominent capitalists of that city, bankers, presidents of trust companies, and railway men. All were impressed with the frank manner and attractive plans of Brice, and they advanced him about five thousand dollars, with which he established a small plant capable of yielding fifteen hundred dollars in gold and silver weekly. Business men of ordinary common sense came to the conclusion that Brice "had either gotten something that is invaluable to any Government—or he is a rank fraud."

Brice himself said to one of these wealthy men:—"We do not want capital, we make our capital here. What we need is Government protection, either in the way of purchase or special legislation; the Government alone can entrust the secret to experts. Our experts would go somewhere to a remote place and make all the gold they wanted as fast as we educated them." When capitalists offered to purchase the secret from Brice he refused, saying the Government alone should have the first option. His claim to obtain gold and silver from chemically pure antimony was examined by Robert W. Hunt, a Chicago chemist, and his favourable report "gave support to the credulous and disconcerted the doubters."

On the 7th of May, 1897, Brice filed an application in the United States Patent Office for a patent on a process for creating gold and silver by operating on lead, tin, antimony, and other base metals. The Patent Office twice refused to grant the claim, on the ground that no practical application of the process had been made; but as Brice continued to press his suit, the officials consented to grant him an experimental demonstration of his methods. Owing to inadequate laboratory facilities in the Patent Office building, application was made to the Secretary of the Treasury for permission to use the complete and spacious laboratory of the Mint Bureau. Accordingly the Secretary of the Treasury requested Mr. R. E. Preston, Director of the Mint, to make a thorough investigation of Brice's process, and three expert assayers were appointed for the purpose. The materials needed for the research were bought of reliable dealers in chemicals by Mr. Preston; they comprised 3 pounds of pure antimony, 2 pounds of rolled sulphur, 1 pound of sheet iron, and some pulverised charcoal. The assay commission made a series of experiments according to Brice's instructions, and three weeks later submitted a report of much interest and value, showing that commercial antimony contained a very small percentage of gold, which was partially recovered by Brice's process. I am indebted to Mr. R. E. Preston, for a copy of this report, which is subjoined.

Public exposure of Brice's scheme for raising money, in the daily press, followed this official report; he himself expressed dissatisfaction with the work of the Commissioners, and still claims that his plant in Chicago is a financial success. Brice's attorney has entered a protest against the findings of the assayers, and here the matter rests.

The circumstances connected with this claim, the appeal to Government authorities, the trial by Officers of the Mint, and the unfavourable report as well as the persistence of the claimant, almost exactly duplicate the events associated with the name of Theodore Tiffereau, in Paris, about forty years ago; and, like Brice, the Frenchman still maintains his discovery, for as recently as December, 1896, Tiffereau addressed a sealed letter to the Academy of Sciences describing a new process to prove that metals are compounds.

These nineteenth century experiences seem worth recording, if only to show how many features they have in common with those of the mediæval chemists,—secret processes, vagueness of description, an assumption of esoteric knowledge expressed in pseudo-philosophic language, personally-conducted experiments with seeming success, and magnificent financial prospects, yet an imperative present need of gold.

(REPORT).

Washington, D.C., May 22nd, 1897.

Hon. R. E. PRESTON,
Director of the Mint,
Washington, D.C.

SIR,—In accordance with your instructions under date of May 3rd, we met at the Mint Bureau on May 5th, to investigate the claim of Mr. E. C. Brice to a process for

producing or creating silver and gold from the base metals, oxides, &c.

Mr. Brice has applied for a patent on this process, and his application has been twice rejected on the ground that the process is inoperative.

We found that you had purchased from the most reputable chemical dealers the materials asked for by Mr. Brice, in a written memorandum, and that the supplies were labelled "chemically pure," as requested by Mr. Brice. A test made by us showed small but weighable quantities of gold and silver in the so-called "chemically pure" antimony. Other samples were procured from different sources with a like result.

While seeking for pure antimony, we accepted the offer of Mr. Brice that he should supervise and direct a trial of his process upon antimony known to contain small amounts of silver and gold, and that he should conduct an assay of the same antimony for a comparison of results, from his own assay methods with those from his "creative process." His assay, in which he scorified one-half assay ton (one assay ton equals 20,166 grms.) with one-half assay ton of lead, showed the antimony to contain 0.066 ounce of gold and 0.317 ounce of silver per ton.

Mr. Brice now subjected five (5) ounces of this antimony to his creative process. His yield, after treatment, showed gold, 0.084 ounce per ton of antimony, and 0.670 ounce silver per ton of antimony used.

Your Committee followed up this work by making an assay of the same metal, following well-known and approved methods of assaying, with the following results:—Gold, 0.100 ounce per ton; and silver, 1.20 ounces per ton of antimony. A comparison of this result will show that Mr. Brice found by his assay sixty-six per cent (66%) of the gold, and twenty-six and forty-one hundredths per cent (26.41%) of the silver actually present in the materials used.

By his "creative process" he recovered eighty-four per cent (84%) of the gold and fifty-five and eighty-four hundredths per cent (55.84%) of the silver originally present in the materials.

It was judged by the Commission that they were not likely to obtain decisive results so long as—working on materials containing appreciable quantities of silver and gold—comparisons would have to be made between minute quantities of those metals found by assay with those obtained by the creative process of Mr. Brice. These differences might be within the limits of error in working, as the amounts obtained were at best very minute, and at times so minute, indeed, that only the most delicate balance in the Institution barely indicated any weight, although this instrument is sensitive to the 1/250th part of a m.grm. (6/100000 grain).

To eliminate the doubt which might thus arise in the minds of those unfamiliar with the limits of accuracy in assaying, it was decided to obtain, if possible, metals entirely free from gold and silver.

The Preparation of Pure Antimony.

We had found all available samples of metallic antimony to contain minute but appreciable quantities of gold and silver. We undertook the task of preparing antimony, which upon assay should not show even a trace of either of those metals. Various methods to attain this end were considered. Distillation of the metal would be obviously ineffective, since in Mint practice it is well known that small amounts of silver and gold are carried off in the vapours of the volatile metals, such as antimony, zinc, &c. An effort was made to prepare pure metal in the wet way from antimony sulphide, but the operation was probably conducted with too much haste, since the resultant metal contained the seemingly inevitable trace of gold and silver. We then had recourse to the process of Capitaine, which is recommended by Gmelin as furnishing the purest antimony (vol. iv., p. 320, published in full in *Journal Pharm.*, xxv., 516). This process consists essentially in

the reduction of metallic antimony from pure double tartrate of antimony and potash. To prevent the reduction of the potash from excess of carbon present, and to form a more fluid melt from which the antimony could settle more perfectly, we added a sufficient amount of pure potassium nitrate to oxidise the excess of carbon present beyond that necessary for the reduction of the oxide of antimony.

The reguline metal thus obtained, amounting to about 20 ounces, was melted under a covering of potassium nitrate. This operation of fusing with the nitrate was repeated until, besides any possible potassium present, a considerable portion of the antimony had been oxidised.

The metal thus obtained, now only some 18 ounces, was finely powdered and treated to a prolonged boiling in distilled water. Not the faintest alkaline reaction was shown by this treatment. We assured ourselves of the absence of gold and silver by repeated assays, and the metal so prepared was used in our subsequent experiments.

Pure Lead.

Lead is used either as metal or oxide in all assaying processes. It is evident therefore that, in this investigation, lead free from gold and silver was equally desirable. It is well known to chemists and assayers that even the so-called "chemically pure" and specially-prepared leads contain traces of these metals. Becker, in his Monograph on the Comstock Lode (*Geological Survey*, 1882), describes the difficulty he encountered in using even the purest leads available, since the trace of gold and silver was so unevenly distributed that he could not make an allowance for the same with any assurance of accurate results. After much time and labour expended in an attempt to prepare lead or litharge free from gold and silver, he was forced to use a lead containing approximately 0.07 ounce of silver per ton. All the leads sold by chemical dealers to-day as "chemically pure," though more free than Mr. Becker's lead from gold and silver, are only comparatively so, since they all show a minute bead of those metals if sufficient quantity be taken for the test.

In the hope of producing a lead which should be absolutely free from gold and silver, we procured a quantity of Squibb's "chemically pure" lead acetate. This was dissolved in distilled water, a few drops of potassium iodide added to produce the most insoluble salt of silver known to us under the circumstances,—and, when thoroughly mixed, a few centimetres of dilute sulphuric acid produced a bulky precipitate of lead sulphate which served the purpose of collecting and carrying down any silver iodide which might have been present. It may not be amiss to mention the fact for the benefit of chemists that this lead sulphate precipitate yielded a notable quantity of silver when reduced.

The acetate solution thus freed as we hoped and supposed from silver, was precipitated by dilute sulphuric acid, the lead sulphate washed with distilled water until free from acid. It was then boiled with a solution of ammonia and ammonium carbonate, until the sulphate was converted into carbonate. This compound, when thoroughly washed so as to be free from ammonia salts, was converted into lead oxide (litharge) by heat. Very much to our surprise, four assay tons of the litharge thus prepared yielded a weighable quantity of gold and silver. In view of the limited time at our disposal, we abandoned further efforts in this direction, and having obtained through Mr. Jacob Eckfeldt, Assayer of the Mint at Philadelphia, a sample of lead, which when two (2) assay tons were used for test, showed no visible bead of silver, we used this lead in our subsequent experiments so long as the limited supply lasted. Having now at our disposal antimony and lead, neither of which showed any silver or gold visible to the eye, when used in reasonable quantity, we proceeded to repeat the "creative process" of Mr. Brice, following strictly his instructions as given by him

in his first exhibition of his process, and as slightly modified by his written communication of May 15th.

For this trial we took two (2) assay tons of our specially prepared antimony, — carried it carefully through each step of the "Brice" process, which necessitated the use of six (6) assay tons of lead for its completion.

The result was entirely negative, no trace of gold or silver being visible.

The experiments thus conducted would seem to dispose effectually of Mr. Brice's claim to any creation of gold and silver. But it was deemed best to invite the claimant with his friends to witness, and he to direct, a repetition of the test. This offer was made, and promptly declined by Mr. Brice when he learned that the result of our test was entirely negative. He, however, insisted on the trial of two other and distinctly different methods, neither of which up to this time had been suggested. We yielded, and were given to understand that these methods were improvements on the original process shown us, and that a much larger production of the precious metals might be expected.

Experiment No. 1. — At the behest of Mr. Brice we weighed out two and one-half assay tons of antimony, and subjected it to treatment under his immediate direction. The result was a regulus weighing three assay tons. Mr. Brice directed that the whole of this regulus should be scorified with nine assay tons of lead, and the final button cupelled. The final result was a minute bead of metal, which, when placed on the delicate balance used, weighed 75/1000 of a milligramme (1/1000 grain).

This bead was treated with nitric acid, and a slight trace of gold remained.

Experiment No. 2. — Two (2) assay tons of antimony were treated by a still different method under the direction of Mr. Brice.

A regulus was obtained weighing four and one-quarter assay tons. This was pulverised and treated by a process in which a new feature was introduced. The resulting ore was divided into two parts, because of the necessity of using two different leads, the remaining six assay tons of that from the Philadelphia Mint serving for only two assay tons of the ore. For the remaining two and one-quarter assay tons we were compelled to use seven assay tons of test lead obtained from Richards and Co., and marked "Strictly free from gold and silver." From neither of these operations comprised in Experiment No. 2 was any visible bead obtained.

The contents of the Richards' lead in silver and gold being an unknown quantity, it was thought prudent to conduct a check assay upon this lead, using seven assay tons for the purpose.

A bead was recovered weighing 25/1000 of a m.grm., which when treated with nitric acid showed a slight trace of gold. Mr. Brice in his operation, using this same lead, failed to recover the silver and gold shown to be present in the materials used by him. The loss of this minute quantity of silver and gold would have no significance but for the statement on the part of Mr. Brice that it was from this variation of his process that he expected his best creative results.

Referring to Experiment No. 1, of May 18th, it should be stated that we subsequently received a further supply of the Philadelphia lead, together with a letter from the firm furnishing it, in which they state that the lead was specially prepared for them, and was the purest known to them. They state that it contains less than 0.02 ounce of silver per ton. An assay made by us upon a large amount of the lead confirms the statement of the Philadelphia firm as to its high degree of purity; yet it contains in nine assay tons sufficient silver and gold to fully account for the minute bead obtained by Mr. Brice in Experiment No. 1 of May 18th.

Conclusion.

During these experiments, which have extended over

some three weeks, and have involved an amount of painstaking labour which we hope has not been entirely wasted, we have seen not the slightest evidence of any "creation" or transmutation.

On the contrary, the claimant failed in every instance to recover the entire amount of silver and gold known to be present in the materials. The claimant seems to have devised a variety of irrational and wasteful methods for recovering a portion of the silver and gold known to metallurgists as being present in many commercial metals, such as antimony and lead.

Respectfully submitted,

(Signed) ANDREW MASON,
Sup't. U.S. Assay Office,
New York.

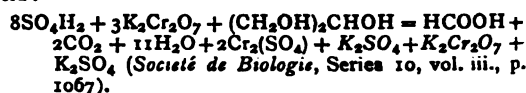
(Signed) D. K. TUTTLE,
Melter and Refiner, U.S. Mint,
Philadelphia.

(Signed) CABELL WHITEHEAD,
Assayer, Bureau of the Mint,
Washington.

ON THE ESTIMATION OF GLYCERIN BY BICHROMATE OF POTASH AND SULPHURIC ACID.

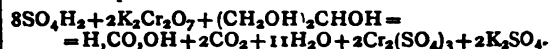
By F. BORDAS and SIG. DE RACZKOWSKI.

We have given the following equation as the formula for the oxidation of glycerin by bichromate and sulphuric acid:—



Chromic acid oxidises glycerin, giving, in every case, carbonic acid, water, formate of chromium, chromate of chromium, formic aldehyd, &c. In the presence of sulphuric acid there is produced, therefore, besides the carbonic acid and the water, *free formic acid* and sulphate of sesquioxide of chromium.

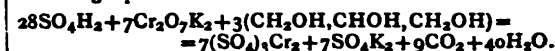
Let us assume the formation of the double salt ($\text{K}_2\text{SO}_4 + \text{K}_2\text{Cr}_2\text{O}_7$) composed of sulphate and bichromate of potash. Now, as this salt will only form when the proportion of sulphuric acid is insufficient to saturate the whole of the potash of the bichromate, we must admit that, as we have an excess of sulphuric acid, the bichromate is entirely decomposed, and therefore this salt cannot be formed under the conditions existing. The amended equation will therefore be—



While in the preceding expression 1 part of glycerin would correspond to 9.62 of bichromate, in the new one it corresponds to only 6.41.

The titration value of the bichromate, of which 1 c.c. should correspond to 1/1000th (the experiment being on 5 c.c. of glycerin solution), which was 48 grms. per litre, now becomes 32 grms. per litre.

In a recent note (*Journ. de Pharm. et de Chim.*, p. 426, May 1, 1897) M. Nicloux asserts that the oxidation of glycerin does not produce formic acid, and he adopts the following equation as the formula of the reaction:—



In this formula the transformation of glycerin into carbonic acid and water is effected *integrally*. He deduces from this that the titration-value of the bichromate solution should be 37.28 per 1000. M. Nicloux then proposes to modify our process by employing a solution of this value.

We could show that M. Nicloux is mistaken in his statements concerning the oxidation of the glycerin by quoting experiments which prove that its decomposition under the influence of bichromate of potash and sulphuric acid *does not proceed integrally*, producing carbonic acid and water, and that there is always undecomposed formic acid present when operating in the way we have described. In fact, the action of chromic acid on formic acid is far from being energetic, seeing that if we heat formic acid with chromic acid, in excess or not, one part decomposes into carbonic acid and water, another part volatilises, and the remainder combines with the sesquioxide of chromium, forming formate of chromium.

It is only after heating for some time in presence of an excess of sulphuric acid, and cooling gradually, that the decomposition is complete.

We prefer, however, not to insist on this point, as discussion is of no interest, given that neither the solution of bichromate of potash at 48 grms. per litre; that of 32 grms. per litre, resulting from the modification of our equation; nor, finally, that of 38 grms. per litre advocated by M. Nicloux, represent definite titration values.

The influence of the proportion of sulphuric acid employed is shown at about one-tenth of a cubic centimetre. It results, therefore, that the titration of the solution of bichromate, being intimately allied to this proportion of sulphuric acid, is absolutely *empirical*, or that the quantity of this solution necessary to obtain the yellowish green tint varies with the quantity of acid, and also with its degree of concentration. This is easily verified by experiment. We can see that, given a solution of bichromate of any titration value, we can determine by successive trials the quantity of acid to use, so that when we use 5 c.c. of glycerin solution, a definite volume of this latter—say, 1 or 2 c.c., for example—will correspond to 18 grms. per litre of glycerin.*

The *modus operandi* proposed by M. Nicloux, which consists of using a solution of 38 grms. per litre in the presence of 4 or even 5 c.c. of pure concentrated sulphuric acid, *boiling if possible*, prevents the grave inconvenience of giving four different results, according as we work with one or the other of the proportions indicated by him, for 4 and 5 c.c. of pure concentrated sulphuric acid, and the same quantities *boiling* constitute four different proportions.

In conclusion, we can only confirm what we have already said (*Comptes Rendus*, Dec. 14, 1896, p. 1072; and *Société de Biologie*, vol. iii., p. 1067), viz., that a solution of pure crystallised bichromate of potash at 24 grms. per litre serves perfectly well; that 2 c.c. of such a solution corresponds to 1 grm. per litre of glycerin, when working on 5 c.c. of glycerin solution and employing *exactly* 2.5 c.c. of pure concentrated sulphuric acid.*

Finally, we would remark that we never pretended to produce a process giving the glycerin to 0.001; for, being colorimetric, there are, of course, difficulties which are inherent to such methods. As we have described it, the process appears to us practicable and able to give results sufficiently near for the purpose for which we devised it; that is to say for the estimation of glycerin in wood ferments.—*Journ. de Pharm. et de Chim.*, Series 6, vol. vi., No. 2.

A NEW AND RAPID METHOD

FOR THE

QUALITATIVE SEPARATION OF IRON, ALUMINIUM, CHROMIUM, MANGANESE, ZINC, NICKEL, AND COBALT.

By ALEXANDER RAMSAY CUSHMAN.

ADD ammonium chloride, ammonia to alkaline reaction and then ammonium sulphide in slight excess. Warm and filter. Wash the precipitate and remove from the filter while still moist. Warm with moderately dilute hydrochloric acid in a porcelain dish. Complete solution of the precipitate shows the absence of nickel and cobalt. If a black residue remains, one or both are present, in which case add aqua regia, and warm gently until dissolved. Expel excess of acid and free chlorine by evaporation, and then make the solution strongly alkaline with ammonia, after previously adding ammonium chloride in case the amount of hydrochloric acid used upon the sulphides was small. Add bromine solution in excess, and allow to stand a few minutes. Filter and wash.

The Table shows the course of the analysis after this point is reached.—*American Chemical Journal*, vol. xix., No. 7.

* It can be conceived that there may be under these conditions an unlimited number of modifications in our method of estimation, taking care, of course, to vary in the same manner both the bichromate solution and the quantity of sulphuric acid to be added.

* We mentioned 2 c.c. of sulphuric acid as being the proportion to use. Now, with this quantity, 2.1 c.c. of solution of bichromate at 24 grms. per litre is necessary to obtain a yellowish green tint; this would cause an error of 0.05 per 1000.

Precipitate 1.— $\text{Fe}_2\text{O}_3, \text{MnO}_2, \text{H}_2\text{O}, \text{Al}_2(\text{OH})_6, \text{Cr}_2(\text{OH})_6$. Remove from the filter and add KOH in excess, then Br solution and filter.

Filtrate 1.— $\text{ZnCl}_2, \text{NH}_4\text{Cl}, \text{NiCl}_2, \text{NH}_4\text{Cl}, [\text{Cl}.\text{Co}(\text{NH}_3)_5]\text{Cl}_2$. Add KOH in large excess, allow to stand a few minutes and filter.

Residue 2.— $\text{Fe}_2\text{O}_3, \text{MnO}_2, \text{H}_2\text{O}$. Divide in 2 parts. Part 1. Dissolve in HCl, add a few drops of NH_4CNS . Blood red colour shows $\text{Fe}_2(\text{CNS})_6$ and proves Fe. Part 2. Fuse with $\text{Na}_2\text{CO}_3 + \text{NaNO}_3$ in O. F. of blowpipe. Bluish green mass proves Mn.

Filtrate 2.— $\text{K}_2\text{Al}_2\text{O}_4, \text{K}_2\text{CrO}_4$. Divide in 2 parts. Part 1. Acidify with HCl, add excess of $(\text{NH}_4)_2\text{CO}_3$ and boil. A white flocculent precipitate shows $\text{Al}_2(\text{OH})_6$ and proves Al. Part 2. Acidify with H_2O_2 and add $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$. A yellow precipitate shows PbCrO_4 and proves Cr.

Precipitate 3.— $\text{Ni}(\text{OH})_2$. (Greenish white). Confirm in salt of phosphorus bead in the R. F. of the blowpipe. Yellow bead, cold, proves Ni.

Filtrate 3.— $[\text{Cl}.\text{Co}(\text{NH}_3)_5]\text{Cl}_2, \text{K}_2\text{O}, \text{ZnO}$. Heat to boiling and filter.

Precipitate 4.— $\text{Co}(\text{OH})_3$. Confirm in borax bead, in the O. F. of the blowpipe. Blue bead proves Co.

Filtrate 4.—Acidify with $\text{H}_2\text{C}_2\text{O}_4$, and saturate with H_2S . A white, flocculent precipitate shows ZnS . Confirm by ignition with $\text{Co}(\text{NO}_3)_2$ on charcoal. Yellow-green mass proves Zn.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, June 17th, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

Mr. Samuel Pollitt was formally admitted a Fellow of the Society.

A Certificate was read for the first time in favour of Mr. Oscar Guttman, 12, Mark Lane, E.C.

The following were duly elected Fellows of the Society:—William Ackroyd; Walter Harry Barlow; William Malam Brothers; Gerald Noël Brown; Ernest Stuart Cameron; Medwin C. Clutterbuck, B.Sc., Ph.D.; William Cranfield; A. Bilderbeck Gomess; Frederick Roscoe Grundey, B.Sc.; Edward Halliwell; Frank William Harbord; Harold Harman; B. J. Harrington, Ph.D.; John Edwin Mackenzie, B.Sc., Ph.D.; William Robertson Pollock; Lionel Walter K. Scargill, B.A.; James Porter Shenton; William Taverner.

Of the following papers those marked * were read:—

*79. "Molecular Refraction of Dissolved Salts and Acids." Part II. By J. H. GLADSTONE, D.Sc., F.R.S., and W. HIBBERT.

The present paper is a continuation of a previous communication to the Society two years ago, under the same title (*Proc.*, 1895, xi., 120). It is especially concerned in replying to the questions, "Has a salt the same molecular refraction whether it be in the crystalline state or in solution?" and "How far is any refraction change dependent on the solvent used?" The paper also gives some conclusions to which the data appear to lead.

In a table previously published (*Trans.*, 1895, lxvii., 831) there were many comparisons between the specific refraction of solid salts and their value in solution, but no crystals were examined excepting those which had only one axis, or where the different indices were very near together. By adopting the method of Damien as suggested by Pope, we now add seventeen more cases having two or three indices of refraction. The observations are in accordance with the conclusions previously drawn, the refraction of the salt in solution being in some cases greater, and in other cases smaller, than that of the crystallised body. The change of refraction, however, rarely if ever, amounts to 4 per cent.

In making experiments on the effect of different solvents we have examined nine salts and acids, including those published in 1870 (*Trans.*, 1870, xxiii., 101). The first result is to show that the specific refraction of the substances dissolved in water does not generally differ much from the value yielded by solution in other solvents. If, however, we examine those substances which show a great change of refraction when deduced from different strengths of solution in water, the result is very striking. A comparison of hydrochloric acid when dissolved in water and in different alcohols and ethers, is shown in a diagram of curves. Whilst in water the specific refraction of the acid is raised in the first instance from about 0.300 to 0.386, and then gradually rises on dilution to 0.400; the acid dissolved in the different alcohols shows a lower starting-point and a more gradual rise, in the following order:—Methyl, ethyl, amyl, and capryl alcohols, followed by a very low starting-point in the case of ethyl ether, with an actual decrease on dilution, and by scarcely any change at all in the case of amyl ether.

In the case of lithium chloride, which gives in water a curve rising on dilution second only to that of hydrochloric acid itself, the solution in alcohol yields a curve very similar in character. Ferric chloride, which is the most striking instance of a great decrease in refraction occurring on dilution with water, shows also a decrease when it is dissolved in alcohol and acetic ether. Nitric acid, when mixed with water, shows a great change of specific re-

fraction, but when dissolved in nitrobenzene shows little if any.

The authors express their growing conviction that neither the salt nor the solvent really changes its specific refraction, but that by their interaction some new product or products result, in quantities determined by the proportionate amount of the two original substances. The action may be one of dissociation or of association, or of some hitherto unrecognised re-distribution. It is the changing proportion of this *tertium quid* which makes itself apparent by the changing specific refraction of the solution.

DISCUSSION.

The PRESIDENT said that the Society was much indebted to Dr. Gladstone for this last addition to his great work on the refractive indices of chemical substances. He wished it were possible to determine directly the refractive index of fluorine, which seemed, from Dr. Gladstone's results with fluorine compounds, to possess the smallest refractive power of any of the elements.

Professor DUNSTAN asked whether the authors had considered the possible influence of the formation of the alkyl chlorides on the solutions of hydrogen chloride in alcohols.

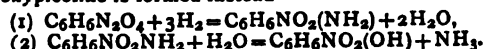
Dr. GLADSTONE, in reply, said they had been unable to detect the formation of any alkyl chloride in the solutions.

*80. "On a Space Formula for Benzene." By J. NORMAN COLLIE, Ph.D., F.R.S. (This paper will appear in our next issue).

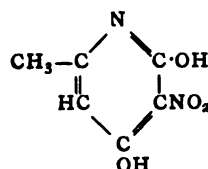
*81. "On the Production of some Nitro- and Amido-Oxypticolines." By A. LAFWORTH, D.Sc., and J. NORMAN COLLIE, Ph.D., F.R.S.

When dioxypicoline (*Trans.*, 1891, 59, 617) is warmed with 60 per cent nitric acid, a nitro-compound is at once produced, $C_6H_7NO_2 + HNO_3 = C_6H_6NO_2 + H_2O$. This nitrodioxypicoline possesses all the properties of a nitrophenol, it is light yellow in colour, and forms salts with bases.

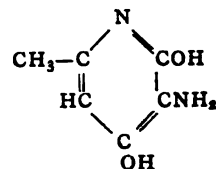
When it is carefully reduced with tin and hydrochloric acid, an amido-oxypticoline results, but should the temperature rise too high a secondary reaction occurs and a trioxypicoline is formed instead—



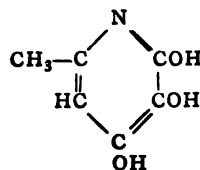
The molecular constitution of these three compounds may be expressed by the formulæ:—



Nitro-compound.



Amido-compound.



Trioxypicoline.

The amido-compound is remarkable for the series of brilliant colours it produces when treated with various oxidising agents; deep indigo-blue with ammonia and air, orange yellow with nitric acid, a deep moss green with alkaline ferricyanide of potassium, and a brilliant magenta with weak acetic acid and potassium bichromate. These colourations are very similar to those produced when various alkaloids are treated in a similar manner.

This amido-oxypicoline forms salts with strong mineral acids but is also capable of liberating carbon dioxide from alkaline carbonates.

When its hydrochloride is only partially neutralised with sodium carbonate and the solution is boiled, an excessively insoluble substance separates, which seems to be a compound of the dioxamidopicoline and the trioxypicoline—



This insoluble compound by persistent boiling with strong hydrochloric acid is finally changed into the tri-oxypicoline and ammonia.

The trioxypicoline may be prepared at once from the dioxamidopicoline hydrochloride, by using the full amount of sodium carbonate necessary to neutralise the hydrochloric acid, and then boiling for half an hour. It then crystallises out in long, needle-shaped crystals.

This trioxypicoline resembles pyrogallol in many of its reactions, it is an excessively powerful reducing agent, and in alkaline solution will develop photographs. It will precipitate silver from a solution containing a considerable quantity of free nitric acid, and it also gives with oxidising agents a series of colour tests similar to those of the dioxamidopicoline.

*82. "Further Experiments on the Absorption of Moisture by Deliquescent Substances." By H. WILSON HAXE, Ph.D.

In a preliminary note (*Proc.*, 1896, 12, 33) the author showed that certain deliquescent salts, when exposed to the air, attained a maximum of hydration, and that its maximum corresponded to a definite number of molecules in a large number of cases.

In the preliminary experiments no reference was made to the vapour-pressure of water in the air, but in experiments since made, the condition of the atmosphere as regards moisture has been carefully noted, or an artificially saturated atmosphere has been contrived under known conditions of temperature.

Having now experimented with 10 deliquescent chlorides (lithium, magnesium, cadmium, calcium, copper, nickel, cobalt, iron, manganese, and platinum), 3 nitrates (sodium, magnesium, and manganese) with sulphuric acid and with sodium formate, under various conditions, it was found that (1) they attract quantities of water corresponding in all cases to a definite hydrate, (2) after deliquescing to a maximum there followed in all cases a decline in weight, and in four instances the salts returned to their original hydration and crystallised out, and that (3) the amount of hydration, though apparently always corresponding to a definite number of molecules of water, is not always the same, but seems to depend within certain limits both on the temperature and the relative humidity of the atmosphere and also on the conditions under which the air has access to the salt.

The author suggests that the above experiments demonstrate the phenomenon of deliquescence to be caused by hydration of the deliquescent salt.

*83. "The Fusion Point, Boiling Point, and Specific Gravity of Nitro-benzene." By R. J. FRISWELL.

Great discrepancies are found in the books as to the above constants.

Schultz gives in his first edition, 1882, 1·2002 at 0° and 1·1866 at 14·4°; in his second edition, 1·208 at 15°. Fusion point +3°.

Beilstein gives the same, and quotes Mitscherlich for the fusion point and Kopp for the specific gravity.

Gmelin gives 1·209 and +3°, and quotes Mitscherlich. As to boiling point, Gmelin, quoting Mitscherlich, gives 213°, Schultz, 1882, gives 210°; in 1886, 206–207°, Beilstein quoting Städeler, 205° at 730 m.m.

It would thus appear that several of these numbers have been quoted unverified for over 60 years.

No statement of specific gravity of solid nitrobenzene has been published excepting that in *Watts' Dictionary*, given to A. G. Green in a private communication by

R. J. Friswell. Determinations of the various points have been made. Calculated for comparison with water at 4° the sp. grs. are:—

	<i>t</i>	<i>d</i>
Solid	1·5	1·3440
Liquid	3·8	1·2220
"	13·0	1·2160
"	28·0	1·1931

Boiling point corrected 209·0°.

Melting and solidifying point +5°.

Nitrobenzene is remarkable as having a distinctly coloured vapour very closely resembling that of chlorine. The colour is easily visible in a thickness of about 2 inches and is strongly marked when 6–8 inches are examined. The author is not aware of any other organic vapour of so simple a constitution which is visibly coloured.

No bands of absorption are shown in the visible spectrum when light is transmitted through this vapour. The violet and blue are absorbed as with the fluid but less strongly.

*84. "The Action of Light on a Solution of Nitrobenzene in Concentrated Sulphuric Acid." By R. J. FRISWELL.

Nitrobenzene is, as has long been known, readily soluble in concentrated sulphuric acid of 1·84 sp. gr. and upwards, but a comparatively small amount of dilution precipitates it and at about 1·7 the solubility is very slight.

When a solution in pure concentrated acid is exposed to light it slowly darkens. When exposed to direct sunlight, the darkening goes on with great rapidity, and in a few minutes the solution becomes quite black and opaque, then the action ceases. The light from burning magnesium produces the same effect.

The solution has been kept unchanged in the dark for upwards of four years. If the exposure to light takes place in a stoppered bottle, a slight odour of sulphurous acid is perceptible after some time in the air above the solution.

If the nitrobenzene thus used is recovered and re-distilled and re-dissolved in sulphuric acid it behaves in exactly the same way.

Attempts were made to increase the change by spreading the solution on glass beads and between sheets of glass, but the depth of the colour of the product soon brought all change to an end.

Several hundred grammes of the black solution were prepared and attempts made to isolate the products of change, but though a brownish calcium salt was obtained and an ammonium salt in solution, the latter decomposed on evaporation with a caramel like odour; what was left was treated with phosphoric chloride, but no satisfactorily pure product could be obtained: the matter needs further investigation.

The rate of 'blackening' of the solution is undoubtedly a measure of the actinic power of the light.

DISCUSSION.

Mr. A. G. GREEN remarked on the possible similarity between the action of sulphuric acid on nitrobenzene in the presence of light, and the electrolytic reduction of a solution of nitrobenzene in sulphuric acid which had been investigated by Gattermann. In this case it was shown that at first phenylhydroxylamine, then paramidophenol was the product.

Dr. HEWITT said that he had noticed that colouration occurred in sulphonating nitrobenzene, even in the dark.

The PRESIDENT drew attention to the fact that in its changes of density near its solidifying point nitrobenzene appeared to show the same peculiarity as water, viz., an enormous change, but in an opposite direction. The action of light on the nitrobenzene solution in sulphuric acid was remarkable. If a flash of magnesium light were permitted to fall on the nitrobenzene solution through a

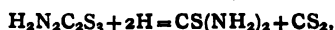
quartz window, he thought that the blackening would furnish a good lecture illustration.

Mr. FRISWELL, in reply, said that he thought it quite possible that the change in the solution of nitrobenzene in sulphuric acid when exposed to light was of the same kind as that induced by electrolysis. He had never observed any colouration on sulphonating nitrobenzene when the materials were pure and light was excluded.

85. "The Reduction of Perthiocyanic Acid." By F. D. CHATTAWAY, M.A., and H. P. STEVENS, B.A.

The atoms forming the molecule of perthiocyanic acid have been assumed to be arranged in a simple ring from the observation that on reduction it yields thiourea and carbon bisulphide. As, however, the properties of the substance render it probable that its molecule is larger than that represented by the simplest possible formula, $H_2N_2C_2S_3$, the conclusion that in this complex molecule all the structural units have a similar atomic arrangement is only valid if the amounts of thiourea and carbon bisulphide obtained on reduction approach those given by theory. The reduction of perthiocyanic acid has therefore been carefully carried out in various ways to determine the exact amounts of carbon bisulphide and thiourea obtainable and the nature and amount of any other products of the action.

When perthiocyanic acid is reduced by tin and hydrochloric acid, carbon bisulphide and thiourea are produced in almost theoretical amount,



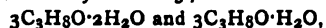
only very small quantities of hydrogen sulphide and carbon dioxide are produced in addition, these being doubtless formed by the hydrolysis of a small portion of the perthiocyanic acid under the influence of the hydrochloric acid, $H_2N_2C_2S_3 + 2H_2O = CS(NH_2)_2 + H_2S + CO_2 + S$.

86. "The So-called Hydrates of Isopropyl Alcohol." By T. E. THORPE, LL.D., F.R.S.

Four hydrates of isopropyl alcohol are stated to exist:—



isolated by Erlenmeyer in 1863;



discovered by Linneman in 1865; and $C_3H_8O \cdot H_2O$, prepared by Ruhemann and Carnegie in 1888. All these hydrates boil within a comparatively small range—from 78° to 81° —whereas the amount of water they contain varies from 9 to 23 per cent.

The author gives reasons for doubting the existence of these hydrates as distinct chemical entities capable of definite isolation. By studying the behaviour of mixtures of isopropyl alcohol and water, it would appear that within certain fairly wide limits, water and the alcohol distil together in indefinite proportions, and that the water tends to pass over more rapidly than the alcohol, and hence is found in the largest proportion in the fractions of lowest boiling point. There is no evidence for the existence of these hydrates at their respective boiling points. Nor is there any more evidence for their existence at ordinary temperatures.

By synthetically forming them by the direct addition of the required amount of water to the alcohol, and allowing them to partially evaporate at the ordinary temperature of the air, it is found that the alcoholic strength of the residue is greatly increased; or, in other words, the water evaporates faster than the alcohol, although the latter boils 20° lower than the former. The two substances, therefore, are not in stable union, even at ordinary temperatures.

When the relative densities of the synthetically formed "hydrates" are plotted in terms of the amount of water they contain, the values are found to lie on what is practically a straight line; or, in other words, the density of the mixture is, within the limits studied, very nearly a linear function of the amount of the constituents.

87. "The Carbohydrates of the Cereal Straws." By C. F. CROSS, E. J. BEVAN, and CLAUD SMITH.

This paper deals with the results of further investigations of the products of acid hydrolysis of the cereal straws and of the celluloses isolated from them, including also the closely related esparto-cellulose. The results confirm those previously communicated (*Trans.*, 1896, 69, 804—118), that the furfural-yielding constituents (furfuroids) are selectively attacked and for the most part (90 per cent) dissolved; also from the exceptionally high numbers for cupric reduction, that they must exist in solution in a fully hydrolysed form (monoses).

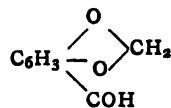
The solutions when neutralised ferment with yeast; carbonic acid and alcohol are produced, and a proportionate effect upon the constants of the solution is shown (density, opticity, cupric reduction, and furfural). Under the conditions, the proportion of the celluloses fermented amounts to 30 per cent of the total dissolved solids. Similar conclusions are deducible from a recent paper by Tollens (*J. für Landw.*, 1897, 106—107), in which the fate of malt-furfuroids in beer-fermentations is discussed. The experimental numbers in this paper show the disappearance of a large proportion of these constituents in the process.

Since the pentoses entirely resist alcoholic fermentation, as shown by Tollens (*Kohlenhydrate*, ii.), and further confirmed by the authors, as well as by later observations of Tollens privately communicated, it is evident that the group of furfuroids thus fermented is constitutionally distinct from the pentoses.

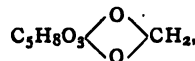
Incidentally to observations on alcoholic fermentation with mixtures of known hexoses and pentoses, the authors find that the latter remain unaffected in presence of hexoses undergoing fermentation. Under certain conditions, however, the pentoses are removed from solution by the yeast organism; the necessary condition appears to be that of "starvation," in the sense, i.e., of the absence of hexoses. The disappearance of the pentose under these conditions is indicated by determinations of furfural and the fall of the furfural numbers. This phenomenon appears to be the simple one of assimilation by the yeast organism, as shown by Bokorny (*Dingl. J.*, 1897, v., 303). The pentose undergoes constitutional change in such assimilation, as the yeast shows no increase in its normal small furfural number.

The authors further discuss the question of the constitution of furfuroids thus shown to yield to alcohol on fermentation, and conclude that the hypotheses of the existence of methylene ethers of the pentoses, or pentose formal, affords, up to the point arrived at, a consistent view of their differentiation from the pentoses.

The history of "piperonal"—



is cited in explanation of the exceptional difficulty of arriving at positive final proof of the analogous constitutional formula—



which sums up the above hypothesis in relation to the group of furfuroids in question. The instability of the pentose as compared with the aromatic residue prevents the application of reactions of resolution (Fittig) or synthesis (Wegscheider) such as have established the methylenic constitution of piperonal.

88. "Studies on the Constitution of Tri-derivatives of Naphthalene No. 16. Conversion of Chloronaphthalene-disulphonic Acids into Dichloronaphthalenesulphonic Acids." By HENRY E. ARMSTRONG and W. P. WYNNE.

In the course of their studies of naphthalene deriva-

tives, the authors have had occasion to make great use of phosphorus pentachloride as an agent for displacing the SO_2H radicle by chlorine. It was therefore necessary to establish in every possible way the validity of this method of determining constitution in the naphthalene series, as it is obvious that the occurrence of isomeric change in any one case would materially weaken the force of all arguments based on its application.

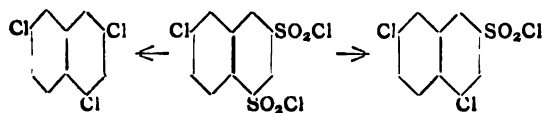
With regard to the nature of the interaction, it is to be noted that in a previous communication (*Proc.*, 1895, xi., 83) it has been shown that it is usually possible to displace with the pentachloride, and to obtain the chloronaphthalene corresponding with the given sulphonie chloride by merely heating the latter alone under appropriate conditions; but that as a rule the chloronaphthalene is obtained somewhat more readily and in larger relative amount when the pentachloride is used. In other words, the main function of the pentachloride is to promote the elimination of SO_2 from the SO_2Cl radicle.

In the case of the chloronaphthalenesulphonic chlorides, the amount of dichloronaphthalene obtained by means of phosphorus pentachloride is relatively considerable, and the residue left after its removal from the crude product by distillation with steam yields nothing but the original chloronaphthalenesulphonic acid on hydrolysis.

The chloronaphthalenedisulphonic chlorides, however, behave somewhat differently, affording but a comparatively poor yield—rarely exceeding 30 per cent of the theoretical amount—of trichloronaphthalene.

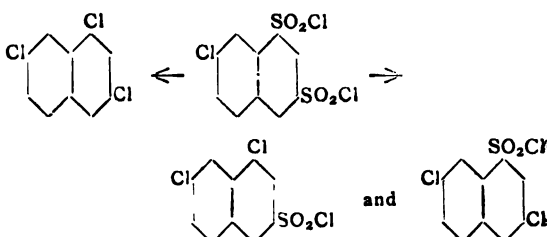
While preparing a full account of their work, the authors have felt it to be incumbent on them to thoroughly examine the residues left after separating the trichloronaphthalenes, which they have had occasion to produce on a large scale (*Proc.*, 1895, xi., 86). Although, in view of the uniformity of the end-products, it was improbable that any change in orientation had taken place at the somewhat high temperatures at which the interactions were effected, it was obviously important to ascertain in every possible way whether such was the fact. The results to be recorded are of interest, as they serve in every case to justify the conclusion previously arrived at, that the treatment of sulphonic chlorides with phosphorus pentachloride may be thoroughly trusted as a means of determining constitution in the naphthalene series. Two cases may be quoted as typical of the behaviour of chloronaphthalene disulphonic chlorides in general.

When 2-chloronaphthalene-4':2'-disulphonic chloride (*Proc.*, 1890, vi., 129) is heated with the theoretical quantity of phosphorus pentachloride at 175° during two hours, it yields both 2:4':2'-trichloronaphthalene and 2:4'-dichloronaphthalene-2'-sulphonic chloride in about equal proportions, about 50 per cent of the material remaining unchanged. 2:4'-Dichloronaphthalene-2'-sulphonic acid affords a sparingly soluble barium salt, crystallising with 34 molecular proportions of water in microscopic needles; a sparingly soluble potassium salt, containing 14 molecular proportions, in thin scales; a chloride crystallising from benzene in small prisms melting at 156° ; an amide crystallising from dilute alcohol in slender needles melting at 166° ; and when the chloride is heated at $180-185^\circ$ with phosphorus pentachloride it is converted into 2:4':2'-trichloronaphthalene. On hydrolysing the chloride in sealed tubes with concentrated muriatic acid at 290° , or the potassium salt with a mixture of sulphuric and phosphoric acids and superheated steam, 2:4'-dichloronaphthalene is obtained. The course of change may therefore be thus represented:—



When 2-chloronaphthalene-1':3'-disulphonic chloride (*Proc.*, 1890, vi., 13) is similarly heated with phosphorus

pentachloride, it yields, besides 2:3':1'-trichloronaphthalene, a somewhat larger proportion of a mixture of 2:1'-dichloronaphthalene-3'-sulphonic and 2:3'-dichloronaphthalene-1'-sulphonic (*Proc.*, 1890, vi., 84) chlorides, about 50 per cent of the material remaining unchanged. 2:1'-Dichloronaphthalene-3'-sulphonic acid, the isomeride present in the larger proportion, yields an anhydrous potassium salt, crystallising in thin elongated scales, but exhibiting a tendency to separate in a gelatinous form; a chloride crystallising from benzene and light petroleum in small prisms melting at 130° ; and an amide crystallising from dilute alcohol in slender needles melting at 218° . When the chloride is heated at $180-185^\circ$ with phosphorus pentachloride, 2:1':3'-trichloronaphthalene is formed. On hydrolysing the chloride in sealed tubes with concentrated muriatic acid at 290° , or the potassium salt mixed with sulphuric and phosphoric acids, in superheated steam, 2:1'-dichloronaphthalene is obtained. The course of change may therefore be thus represented:—



The other α -disulphonic chlorides behave similarly, the tendency being, however, as in the first of the above instances, to form only one of the two possible isomeric dichloronaphthalenesulphonic chlorides, no doubt because the SO_2 of the SO_2Cl radicle, like the SO_2H radicle, is more easily displaced from α - than from β -positions. It is not certain that these products are intermediate in the strict sense of the term, as the effect of prolonging the heating with phosphorus pentachloride at the minimum temperature at which the reaction takes place serves only to increase the yield both of the dichloro- and trichloro-derivatives. As the dichloronaphthalenesulphonic chlorides produced in these interactions decompose at temperatures a few degrees higher— 10° to 15° in most cases—than those at which the corresponding chloronaphthalenedisulphonic chlorides from which they are obtained undergo change, it is not difficult to understand why they escape attack by phosphorus pentachloride under the conditions observed.

89. "Conversion of 1:1' into 1:4'-Dichloronaphthalene by Hydrogen Chloride. The Products of Hydrolysis of 1:1'-Dichloronaphthalene-3-sulphonic Acid." By HENRY E. ARMSTRONG and W. P. WYNNE.

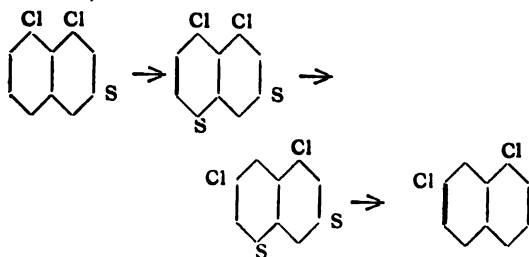
When 1:1'-dichloronaphthalene is heated with concentrated muriatic acid at 290° , it is wholly converted, save for a trace of carbonisation, into the isomeric 1:4'-dichloronaphthalene. This remarkable isomeric change does not seem to occur at temperatures below 200° , but is noticeable at 250° , and complete at 290° ; it does not occur when 1:1'-dichloronaphthalene is heated either alone, or with water, or with concentrated phosphoric acid at 300° , but does happen when it is heated with sulphuric acid of a strength to cause considerable carbonisation. None of the isomeric dichloronaphthalenes show any tendency to change under these conditions.

The experiments which led to these results were made in consequence of the perplexing behaviour of 1:1'-dichloronaphthalene-3-sulphonic acid on hydrolysis. The isomeric α -sulphonic acid (*Proc.*, 1890, vi., 81) requires only a temperature of 230° to effect its hydrolysis, and gives only 1:1'-dichloronaphthalene, whatever be the hydrolytic agent used; the β -sulphonic acid, on the contrary, is not hydrolysed below 285° , and according to the

agent used gives one or other of no less than three dichloronaphthalenes.

1 : 1'-Dichloronaphthalene-3-sulphonic acid is obtained in addition to about an equal proportion of 1 : 1' : 3-trichloronaphthalene when 1-chloronaphthalene-1' : 3-disulphonic chloride (*Proc.*, 1890, vi., 16) is heated with phosphorus pentachloride at 160° (compare preceding abstract). It forms a sparingly soluble anhydrous potassium salt crystallising in thin elongated scales; a chloride crystallising from benzene in thin scales melting at 158°; an amide crystallising from dilute alcohol in short slender needles melting at 197°; and 1 : 1' : 3-trichloronaphthalene when its chloride is heated either with phosphorus pentachloride at 170°, or alone at 209–230°. On hydrolysing the potassium salt with dilute acids, such as 1 per cent sulphuric acid or 50 per cent phosphoric acid at 290°, about 5–10 per cent of the theoretical quantity of 1 : 1'-dichloronaphthalene is obtained, the residue being unchanged salt—a result by which the constitution of the acid is determined beyond doubt. When heated with 5 per cent sulphuric acid or 60 per cent phosphoric acid, carbonisation largely occurs, and with these and stronger acids a small amount of 1 : 4'-dichloronaphthalene is the only substance obtained, a better yield—some 20 per cent of the theoretical—being got when the chloride is heated with concentrated muriatic acid at 290°. The production of 1 : 4' instead of the expected 1 : 1'-dichloronaphthalene under these conditions is to be referred to the action of hydrogen chloride, either present or formed during the carbonisation of the salt.

On effecting hydrolysis by heating the potassium salt, mixed with sulphuric and phosphoric acids, in superheated steam instead of in sealed tubes, an unexpected result was obtained, pure 1 : 2'-dichloronaphthalene, to the extent of 40 per cent of the theoretical amount, being the product, the remainder of the salt being carbonised. The explanation of this change has yet to be given. It is certain that the 1 : 2'-compound is not an intermediate step in the conversion of 1 : 1' into 1 : 4'-dichloronaphthalene during hydrolysis in sealed tubes, both because it is unaffected by prolonged heating with concentrated muriatic acid, and because 1 : 2'-dichloronaphthalene-3-sulphonic acid cannot be detected in the material which has escaped hydrolysis, and, moreover, behaves normally on hydrolysis (compare preceding abstract). It is possible that, under the conditions specified, further sulphonation may precede hydrolysis, and that in consequence of the transference of chlorine to the para-position being thereby prevented, 1 : 2'-dichloronaphthalene is formed, thus:—



Further experiments are being made to test this view.

Of the trichloro-naphthalenes the 1 : 2 : 8 modification is the only one which undergoes change when heated with concentrated muriatic acid. Its sulphonic and disulphonic acids behave similarly, but the course of the action has not yet been worked out, owing to want of material.

(To be continued).

Action of Tannin and Gallic Acid upon the Quino-leic Bases.—Oechsner de Coniack.—The bases behave with tannin and gallic acid not only like the pyridic bases, but like pyridic hydrides.—*Comptes Rendus*, cxv., No. 1.

NOTICES OF BOOKS.

Reform of Chemical and Physical Calculations. By C. J. J. HANSEN, C.E. Printed by the Carlsberg Foundation in Copenhagen. London: E. and F. N. Spon. New York: Spon and Chamberlain. 1897. Pp. 72.

THE deductions contained in this volume are based upon the natural laws of atomic combination, heating, expansion, and compression of æriform substances, and upon the fact discovered by the author—that near the 41° of latitude the specific gravity of oxygen gas of atmospheric density, at the temperature of melting ice, is exactly 1/700th of the gravity of distilled water at its temperature of greatest density.

In different text-books there is great want of uniformity in the values given for many specific gravities, specific heats, &c. (we know one standard work wherein are given no fewer than six values of 1 grm. in grains). This is perhaps because some authors reduce their weights to sea-level, while others take the latitude and elevation of whatever town they may be in. The proposed reform is to reduce all chemical and physical calculations to one common starting-point, from which they can all be fixed by calculation. The proper unit of gravity is hydrogen, but owing to the difficulty in accurately weighing this gas it is better to calculate it from oxygen and nitrogen, both of which have been determined with great accuracy. As all astronomers use the longitude of Greenwich, so, says the author, should all chemists adopt a common circle of latitude, to which all calculations of gravity should be reduced. At a certain latitude, between 41° and 42°, the weight of 1 cubic metre of oxygen at the mean atmospheric pressure and 0° C. is 1017 kilograms. This latitude the author proposes to call the *circle of international gravity*. At this latitude the velocity of falling bodies during the first second is 4.90215 metres, and the length of a pendulum making one oscillation per second = 0.993181 metre. A large number of values of various kinds are calculated and given, such as the *international* atmospheric pressure, boiling-point, specific heats, relative heats, expansion and compression of gases, &c., &c. But there is one important point with which we cannot agree, the author wishes to do away with decimals and go back to the dark ages of vulgar fractions. Some of his calculations done in this manner have a weird and forbidding aspect. Think of it! in adding, subtracting, or multiplying to have to deal with such terminals as 64/185ths, 6/37ths, 2/7ths, 25/37ths, 112/111ths, 867/140ths. These all come in one short calculation of the calories of sensible heat generated by the combustion of 1 kilogram. of Dowson's gas.

There is a certain amount of fascination in Mr. Hansen's idea, but we think it is extremely improbable that such a *bouleversement* will be adopted; it cannot be done gradually, and it is certain that even if it were largely adopted, it would not be universal, and we should then have confusion worse confounded.

Electrolytic Quantitative Analysis. ("Quantitative Analyse durch Elektrolyse.") By Dr. ALEXANDER CLASSEN. Fourth Edition, with 74 Illustrations and 6 Plates. Pp. 249. Berlin: Julius Springer. 1897.

THE fourth edition of Dr. Classen's excellent work will be welcomed by chemists. Electricity and chemistry combined are making such rapid strides of late years, as well in the laboratory and assay office as in the metallurgical workshop, that it is only by frequent editions of such books as this that we are enabled to keep *au fait* with what is latest and newest in the methods employed. It is hardly necessary to go through the book *seriatim*; suffice it to say that Dr. Classen begins at the beginning, both with regard to the theoretical and practical side of

the question, and gradually leads the reader up to his own ingenious and refined methods of electro-chemical analysis. The special bits of apparatus he has designed for this work are all described and illustrated, and the best methods of manipulation and procedure with all the metals, and their electrolytic separation one from another, are given at length. Among the six plates at the end of the book we find a contrast between the old purely chemical laboratory and the modern electro-chemical laboratory. Like so many foreign books, it labours under the disadvantage of not having an Index, which is hardly made up for by an excellent table of contents.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 1, July 5, 1897.

Nomination.—In nominating a Foreign Associate *vice* the late M. Tchebicheff, Prof. Virchow obtained an absolute majority of votes (32). Prof. Stokes, the second candidate on the list, received nine votes.

Thermic Mercurial Ammeter.—Charles Carmichel.—The author has the honour of submitting to the Academy novel mercurial ammeters and voltmeters.

New Mercurial Pump without Cocks and Valves. H. Hanriot.—This paper requires the accompanying illustration.

Action of Tellurium Chloride and Fluoride upon the corresponding Hydracids.—R. Metzner.

Reduction of Molybdenum Anhydride by Hydrogen.—M. Guichard.—The reduction of the oxide MoO_3 below 470° is continuous, and leads directly to the intermediate oxides Mo_2O_5 or Mo_2O_{12} . These intermediate oxides cannot be obtained by means of hydrogen. The author promises to give the results of his experiments on the reduction of molybdc anhydride by means of hydrogen at temperatures exceeding 470° .

On Manganomolybdate.—E. Péchard.—There do not seem to exist compounds for the other acids which, like tungstic acid, yield complex acids.

On Veratrylene-Diamine.—Ch. Moureu.

On Paraxylyl-Acetic Acid and Dimethyl-Phenethyl-oic-2 Acid.—M. Guerbet.

A Novel Carbohydrate, Carubine.—Jean Effront.—Carubine is widely diffused in nature. We have detected it in oats and barley, and it is probably present in beer.

Fermentations in Compound Mediums of Solid Particles.—Th. Schlössing, jun.—The author concludes that aëration, even without agitation, does not affect the cause of fermentation.

No. 2, July 12, 1897.

The Secretary announced the death of Dr. Steenstrup, a corresponding member of the Section of Anatomy and Zoology, which took place on June 20th.

Election.—M. Goyon was elected a Correspondent for the Section of Rural Economy, *vice* the late M. Hellriegel.

Use of Cupric Salts to Prepare for the Determination of various Elements in Cast-irons and Steels.—MM. Ad. Carnot and Goutal.

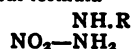
Complexity of the Sheaf of X Rays.—MM. A. Imbert and Bertin-Sans.—A practical conclusion to be drawn from the phenomena observed is, that to obtain a good radiograph, *i.e.*, a proof presenting much contrast, we must

make use of a tube which is still far from being resistant. On the contrary, to effect the radioscopy of a dense medium we must utilise the vacuum at the moment when the less absorbable X rays are present in a sufficient quantity.

On various Basic Copper Salts, and on Brown Cupric Hydrate.—Paul Sabatier.

Reduction of Molybdenum Anhydride by Hydrogen, and the Preparation of Pure Molybdenum.—M. Guichard.—We have established by experiments made at a constant temperature that, in hydrogen between 300° and 470° , the reduction of molybdc anhydride leads to the oxide MoO_2 without passing through the oxides Mo_2O_5 and Mo_2O_{12} .

Action of Benzoyl Chloride upon the Mono-substituted Orthodiamines.—Fernand Mattalet.—The author undertakes to examine what is the action of benzoyl chloride upon the mono-substituted orthodiamines answering to the general formula—



in which the radicle R may be either fatty or aromatic.

Formation of the Mixed Hydrates of Acetylene and some other Gases.—MM. de Forcrand and Sully Thomas.—This paper is not adapted for useful abstraction.

Action of Sulphuric Acid upon Lavoterebenthenes.—G. Bouchardat and J. Lafont.—Among the liquid products of the reaction there has been found fenchene, $\text{C}_{10}\text{H}_{16}$, derived from fenchol by dehydration.

Development of Aromatic Principles by Fermentation in presence of certain Leaves.—Georges Jacquemin.—To prevent the loss of the volatile aromatic principles the gases of fermentation should be passed through a condenser charged with alcohol.

New Hydrolytic Enzyme—"Caroubinase."—J. Effront.—Caroubinase acts energetically at 40° , and its action increases with the temperature up to 50° . At 70° the action becomes very feeble, and at 80° the enzyme is destroyed.

Optical Analysis of Urine: Diabetic Sugar Thermooptically Positive and Negative.—F. Landalgh.

Composition of Haricots, Lentils, and Peas.—Egyptian lentils, as well as beans of the same origin, are the richest in nitrogen. Those of Auvergne are more nitrogenous than those of Bohemia, Spain, Moravia, and Russia. Immature peas are more nitrogenous than those gathered at full maturity.

Revue Générale des Sciences Pures et Appliquées.

No. 11, June 15, 1897.

On Gas and Petroleum Engines.—Aimé Witz.—This long and interesting article is more suited to an engineering than a chemical paper, but it will well repay perusal by chemical engineers. The history of the gas-engine, which is claimed as a French invention, is traced from the commencement in 1799 to the present time, and a number of illustrations are given.

Liquefaction of Fluorine.—H. Moissan and J. Dewar.—Already inserted in full. In a footnote by the editor the readers are informed that the experiments described in this paper were carried out in the laboratory, and with the resources, of the Royal Institution of Great Britain.

The Dinosauriens.—A. Bigot.—Not suitable for abstraction.

The Cultivation of Cocoa in the French Colonies.—H. Lecompte.—Cocoa was originally found only in Mexico, but has since been introduced into, and found in, many other countries. The largest quantity is produced in Brazil and the English possessions in Central America, where the production is steadily increasing; that in the French colonies remains stationary.

No. 12, June 30, 1897.

This number contains no original matter of chemical interest.

No. 13, June 15, 1897.

On X Rays and Dissociation.—Ch. Guillaume.—The idea of dissociation put forth by Sainte-Claire Deville was familiar to spectroscopists, who found therein an explanation of the identity of the spectra of salts, or of a base, under varying conditions. The author claims that it is proved that dissociation is also caused by X rays. When acting on the flesh the ions cause attenuation of virus, excitation of muscle, and vascularisation. The physiological actions of X rays and of currents of high frequency have not merely a chance analogy, the ultimate cause is the same in either case.

On Cape Diamonds.—L. De Launay.—This paper, which describes the Kimberley Diamond Mines and the mining industry, does not materially differ from the Lecture delivered by Sir William Crookes last year at the Imperial Institute on the same subject, but we can find no acknowledgment of the source.

Applications of Electricity to Artillery.—G. Lavergne.—Not suitable for abstraction in these columns.

MISCELLANEOUS.

Reagents, Reactions, Methods, and Formulæ.—The Editor of the *Pharmaceutical Journal* proposes republishing in book form the list of "Reagents, Reactions, Methods, and Formulæ, known by the Names of their Authors," which has appeared in the *Pharmaceutical Review*, and has since been published as a pamphlet. In order to make the list as complete as possible he will be glad to receive from their authors particulars of any tests or processes associated with the names of any chemists. With the idea of increasing its utility the list is now extended by the addition of particulars regarding a large number of microscopical and bacteriological methods and formulæ from the works of Lee, Squire, Crookshank, and others.

The Yorkshire College, Leeds.—The report of the work of the Textile Industries, Dyeing, and Art Classes of this College, shows that in the Session 1896-7 there has been a slight increase in the number of students, and that satisfactory progress has been made in both the theoretical and experimental work. The teaching is being constantly improved and expanded, to keep up with the developments constantly taking place in the weaving industries. During the last five of six sessions there has been a constant and satisfactory increase of senior students. A twelve horse-power gas-engine has been put down since the last session, and it is now possible to run all the machinery, looms, &c., of the department at one time. It is proposed to add a Carding and Spinning Department, and the draft plans have been passed and are now in the hands of the architect. They will provide ample room for a complete plant of scouring, carding, combing, and spinning machinery for both woollen and worsted yarns, and also suitable rooms for conditioning purposes, and for warping, beaming, and sizing machinery.

Carbide of Calcium and the Petroleum Acts.—In view of the growing importance of carbide of calcium, this substance has, by Order in Council, been brought under the provisions of the Petroleum Acts, 1871 to 1881, and it is now necessary for all users of carbide of calcium to obtain from the local authorities under the Petroleum Acts a license to store it. The Acetylene Illuminating Co., Limited, has been manufacturing carbide of calcium since the spring of 1895, and has always laid the greatest stress on the necessity for proceeding with caution in developing this new industry. When proper precautions are taken, there is no more risk attending the use of acetylene gas than there is with coal-gas. As manufacturers of carbide

of calcium, the above-named company are of opinion that the legislative restrictions will not prove a hardship, and they prefer to see this new industry on a well-defined, instead of on an uncertain, basis. Carbide of calcium in itself is, as is well-known, non-explosive; but when water is added to it, or if it be exposed to a damp atmosphere, acetylene gas is evolved, and if this gas comes in contact with a light an explosion will naturally follow. Its presence is, however, easy to detect on account of its pungent odour. The authorities very properly require that carbide should be "commercially pure"; that is to say, it must not contain any impurities liable to generate siliciuretted or phosphoretted hydrogen, which might render the gas spontaneously explosive. Great stress is laid on the importance of not allowing carbide of calcium or acetylene to come in contact with copper, on account of the ease with which acetylide of copper is formed—this body being highly explosive, a touch being enough to set it off. The Fire Insurance Companies require that carbide, liquid acetylene, or acetylene gas must be stored in a separate building, at least ten feet from any other building, and a safety valve must be fitted to the reservoir or pipes, which will allow the free escape of the gas, outside the building, when the pressure exceeds 4 ozs. to the square inch. We have mentioned a few of the more prominent rules which have been proposed, but they are all in course of revision, it being considered that some are too lax, while others are too stringent; it may, however, be well to emphasize the fact that with ordinary precautions there is no more danger than with coal-gas.

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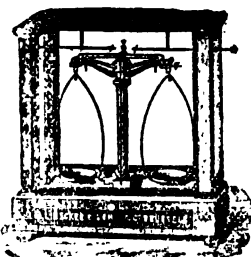
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Fig. 11. No. 91.

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Advertisements for this Number should reach the Office not later than Wednesday, 8th Sept.

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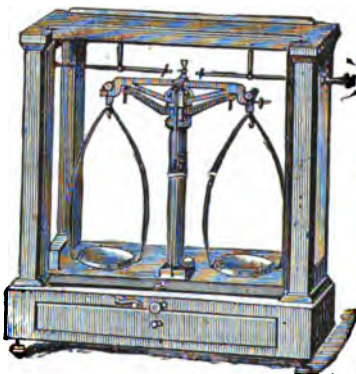


FIG. 3a.

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ON THE EXPLANATION OF A PHENOMENON ATTRIBUTED TO A MAGNETIC DEVIATION OF THE X RAYS.

By Sir G. G. STOKES.

In the *Comptes Rendus* for July 15, 1897 (p. 17), there appeared a note by M. G. de Metz, in which he described an experiment of which the result, according to him, could only be explained by one or the other of these two hypotheses:—Either the X rays are capable of magnetic deviation in an extremely high vacuum, or the cathodic rays are able to traverse the glass envelope of a Crookes tube. I do not think that either of these alternatives contains the true explanation, and I beg the Academy to allow me to submit what is, in my mind, the true explanation of the phenomenon. Everything tends to prove that the X rays are a vibration of the ether, and we may to-day consider it as practically established that this vibration is transversal. If these rays are a vibration of the ether, to suppose that they are capable of magnetic deviation opens up great theoretical difficulties; but, apart from this, I am not aware that such a deviation has ever been demonstrated experimentally. As for the so-called cathodic rays, it seems to me absolutely certain that they are not true rays at all, but simply currents of molecules charged with electricity, thrown off by the cathode. There would be, without doubt, a great difficulty from this point of view, if we were obliged to imagine these molecules capable of passing through the glass of a Crookes tube; the more so, that Crookes himself (*Phil. Trans.*, p. 150, 1879) showed long ago that the cathode rays are stopped by a thin screen of glass, quartz, or mica. But it is in no way necessary to have recourse to this supposition to explain the results obtained by M. de Metz. It seems evident to me that the phenomena which are observed in very high vacua are of the nature of those which have been examined by Spottiswoode and Moulton under the name of *relief-effect* (*Phil. Trans.*, p. 177, 1879). The masses of extremely rarefied air, situated respectively in the Crookes tube and in the cylindrical tube, constitute the two surfaces of a Leyden jar, of which the dielectric is formed by that portion of the envelope of the Crookes tube corresponding with the contour of the cylindrical tube. At each discharge of the induction coil a torrent of negatively electrified molecules is projected against the anti-cathode, or the first surface of the dielectric, which communicates its charge, or a great part of it, either directly to the anode, or, in the first place, to some other part of the internal surface of the Crookes tube. Each momentary charge of the first surface of the dielectric acts inductively on the contents of the cylindrical tube, and produces reciprocally a discharge between the second surface of the dielectric and the aluminium cylinder connected to earth; and in this phase of the reciprocal discharge, where the second surface acts as the cathode, the molecules are projected from this second surface, exactly as from the cathode of the Crookes tube, and they affect a platino-cyanide of barium screen in the same manner.

Although, as I am firmly convinced,—and I believe the greater number of physicists agree with me,—the cathodic rays and the X rays are of a completely different nature, they are both equally capable of affecting a photographic plate or of exciting the fluorescence of a screen of platino-cyanide of barium. This being admitted, the results ob-

tained by M. de Metz are capable of a very simple explanation. When the air in the interior of the cylindrical tube was at the atmospheric pressure, or only at a moderate vacuum, the fluorescence observed was due to the X rays. For, as Lenard has shown (*Wiedemann's Annalen*, li., p. 225, 1894), the cathodic rays—supposing they exist—would be promptly stopped by the air, and would not therefore reach the screen; consequently, the rays producing the fluorescence were found to be insensible to the magnet. On the other hand, at a high vacuum, the cathodic rays, set up by the molecules thrown off by the surface which was made cathodic by induction, were able to reach the screen; and as they were also able to excite a fluorescence much more intense than the X rays, the effect observed was principally due to the cathodic rays; and it is thus that the exciting rays were found to be susceptible of deviation by the magnet.

In offering this explanation, I wish to protect myself against the idea, which might be attributed to me, of explaining in the same manner the apparition of the cathodic rays coming from the second surface of a sheet of aluminium of which the first surface receives the cathodic rays. In this case the *processus* is probably more direct, and presents—to my mind—some analogy with electrolysis.—*Comptes Rendus*, cxzv., No. 4.

ARTIFICIAL LIGHT: MODERN METHODS COMPARED—ELECTRIC- INCANDESCENT, WELSBACH, ACETYLENE.*

By Prof. D. S. JACOBUS.

At the commencement of this Lecture experiments were made to show the appearance of various colours when under different lights. The two sets of lights to be compared were placed about 6 feet apart, and screens were arranged to shade the audience from the direct glare of the lamps. The colours to be examined were on large sheets of cardboard, bent down the middle to a convenient angle, the apex towards the audience; this being placed between the lamps to be compared, allowed each surface to be illuminated by a different set of lamps. Many colours appeared of a different shade when viewed by the acetylene and the Welsbach lights; the lighter shades of pink especially appeared brighter under the acetylene than under the Welsbach light; but the reverse is the case for some other colours. Ordinary gas, and the incandescent electric light also, produced more lifelike tints when the experiment was made of holding the hand between them and a Welsbach lamp.

In a table of the comparative illuminating-power of water-gas and acetylene-gas, we see that acetylene gives ten times as much light as ordinary illuminative water-gas when burned in a flat-flame burner, and about three times as much when the latter is burned in a Welsbach burner. The percentage of the total heat of combustion of the gas, transformed into light, is also greater for the acetylene than for the ordinary illuminating gas. For a given illumination the atmosphere is vitiated by the carbonic acid formed in burning the acetylene, to a slightly less extent than when burning ordinary illuminating gas in a Welsbach burner.

If 3 per cent of acetylene be mixed with air an explosive mixture is produced. In the case of hydrogen 5 per cent is required, and for coal-gas 8 per cent. The maximum amount of acetylene that can be mixed with air to form an explosive mixture is 82 per cent. It therefore appears that acetylene is more explosive than either hydrogen or coal-gas; but, as the burners used for acetylene would discharge a less amount of gas—say one-fifth that of an

* From a Lecture delivered before the Franklin Institute, March 12th, 1897.

ordinary gas-burner—the accidental opening of a burner would cause the atmosphere of the room, as a whole, to be much less contaminated, in a given time, than with coal-gas. On the other hand, acetylene-gas is so much more nearly of the density of air than ordinary illuminating gas that it will not be diffused as rapidly through the air in case of leakage, and will have a greater tendency to collect in a partially enclosed space, and thus cause an explosion in case the gas were ignited.

According to Berthelot, if gaseous acetylene, under a pressure of about 15 pounds above the atmosphere, or over, be ignited by a spark, or by a heated platinum wire, it will decompose explosively without the presence of air, the explosion becoming more violent as the initial pressure is increased. In exploding, the carbon and hydrogen are dissociated, and the heat absorbed in the original formation is set free. This heat, according to Berthelot, is sufficient to increase the temperature about 5000° F. (provided the pressure be kept constant), or to increase the pressure eleven times if the volume be kept constant.

Berthelot likewise found that liquid acetylene, as transported in tanks under pressure, would explode as readily as the gas. He also made experiments to determine if blows or shocks would cause explosions in cylinders of the liquid, and he found that a shock would not of itself start an explosion, but that when steel cylinders of the liquid acetylene were smashed the blow was usually followed by an explosion, probably produced by the sparks generated by the friction of the pieces of broken steel; the sudden opening of a stop-cock may, he thinks, cause local heating, and thus an explosion.

These investigations show how dangerous acetylene is when stored under pressure greater than that of the atmosphere. That such is the case is borne out by the fact that a number of accidents, accompanied by loss of life, have occurred where it has been used under such conditions.

The cost per hour for the production of 16-candle power of light in New York is as follows:—

Incandescent electric light	1'0 cent.
Illuminating water-gas.	Ordinary 4 ft. burner at 1'25 dollars per 1000 cubic feet. 0'5 "
	Welsbach 1'14 ft. burner at 1'25 dollars per 1000 cub. ft. 0'17 "
Acetylene employed for municipal distribution.	Calcium carbide, converted into gas at 40 dollars per ton, to replace ordinary burners 0'5 "
	Calcium carbide, converted into gas at 19'50 dols. per ton, to replace Welsbach, cost of renewing mantles included 0'17 "

It will be seen from this table that, to compete with an ordinary illuminating water-gas, selling at 1'25 dollars per 1000 feet, the carbide would have to be furnished to the gas-company and converted into gas for 40 dollars per ton, to make the same profit and to be as economical to the consumer as ordinary illuminating gas in a flat-flame burner. To be as economical as ordinary gas burned in Welsbach burners, it would have to be made for 19'50 dollars per ton.

The figures for the acetylene are obtained in the following way:—Assume a plant which is furnishing an illuminating water-gas at 1'25 dollars per 1000 feet. If this plant were converted to an acetylene plant, there are certain expenses—such as cost of distribution, office expenses, &c.—which would remain the same: these constant expenses would be practically all, except the cost of making and storing the gas. To simplify matters, let us consider a plant of, say, 500,000 feet of gas per diem. Then we have—

Paid by consumers per day, 500,000 feet at 1'25 dollars per 1000 feet .. 625'00 dollars.
Cost of gas in holder at 40 cents per 1000 feet 200'00 "

Constant daily expenses, together with profit of gas 425'00 "

To supply the consumers with the same amount of light with the flat-flame burners would require one-tenth the volume of acetylene,—hence 50,000 cubic feet of acetylene would have to be stored in the holder for 200 dollars in order that there might be the same amount of profit for the company. This 50,000 feet would be produced by 5 tons of carbide; hence the cost of 1 ton of carbide, together with making the gas, would have to be 40 dollars to compete with ordinary gas at 1'25 dollars per 1000 feet burned in flat flame burners. In the same way it is calculated that to compete with the Welsbach the calcium carbide must be supplied and converted into gas at about 19'50 dollars per ton.

In some of the earlier literature on acetylene, it was proposed to convert electric-lighting plants into plants for the production of acetylene, and thus obtain economic results. This could not be done economically, for only about half as much light could be obtained from the acetylene as by using the electricity direct for incandescent lamps. The results obtained at Spray show that 2 cubic feet of acetylene could be produced per hour per electrical horse power. This would furnish 80 candle power. If the electricity were used direct with incandescent lamps requiring 4½ watts per candle power, the light produced would be 166 candle power per electrical horse power, or about twice as much light as would be produced by the acetylene. To be as cheap an illuminant, therefore, as electricity, the carbide must be made by some means, say water power, where the cost of power and attendance is much less than at the electric lighting station. It has been shown that for equal illumination the incandescent electric light costs twice as much as gas, when the latter is burned in flat-flame burners. The electric light has, however, held its own against gas on account of its superior qualities; that it has done so at a higher cost to the consumer for a given candle power, is proof that other elements enter into the problem of artificial lighting as strongly as the cost of a given amount of light. From this standpoint it may be argued that acetylene, producing as it does a more brilliant light than any now used for interior lighting, and having the quality of showing the complexion in life-like tints, will have its own field, even should it be the most costly style of illumination.

The whole situation may be summed up by saying that each system of lighting has its own field of usefulness, on account of properties peculiar to itself, which make it more desirable than the others for certain classes of work.

CALCULATION OF THE COEFFICIENTS OF EXPANSION OF GASES FOUNDED ON MY THEORY OF VALENCE.*

By JOACHIM SPERBER.

ACCORDING to Gay-Lussac's law all gases and vapours, *mutatis mutandis*, expand equally for an equal rise of temperature, and indeed for 1° by 0'00366 of their original volume at 0°.

In gases of equal relative heat we may always proceed from volumes in which 1° is at the same time 1 cal.

The relative heat of air, hydrogen, oxygen, and nitrogen is approximately that for which 1 cubic metre is in the mean 0'30726; the relative heats of chlorine and bromine

* A Reprint from the *Zeitschrift Anorg. Chem.*

vapour are approximately equal, being in the mean 0.38812 per cubic metre in the average.

Hence it follows that in the former gases the volume is 3.254 cubic metres, and in the latter, for 2.576 cubic metres, 1° is at the same time 1 cal.

The author gives the expansion in the case of those elements whose dissociation- and combining-heats have been calculated.

I. *Fluorine*.—In the chemical dissociation of fluorine the amplitude of the fluorine atoms is extended by 0.0266, for which a dissociation-heat of 87.3 cal. per gramme-atom is needed.

II. *Chlorine*.—In the chemical dissociation of chlorine the amplitude of the chlorine atoms is extended by 0.0134 if a dissociation-heat of 44 cal. per gramme-atom is required.

III. *Bromine*.—In the chemical dissociation of bromine the amplitude of the bromine atoms is extended by 0.005, requiring a dissociation-heat of 16.4 cal. per gramme-atom.

IV. *Oxygen*.—In the chemical dissociation of oxygen the amplitude of the oxygen atoms is extended by 0.005, requiring a dissociation-heat of 83.9 cal. per equivalent, or 167.8 calories per gramme-atom.

The expansions of oxygen and chlorine are inversely as the relative heats of these elements.

The expansions of the amplitudes of the atoms of these various elements per calorie coincide up to the sixth decimal and amount to a mean of 0.000304 (6). This number may be suitably named the linear atomistic coefficient of expansion.

Direct determinations have yielded 0.00366, as is well known.

Our computations refer purely to diatomic gases, in which both the atomic weights and the molecular weights are the weights of equal volumes.

FIFTEENTH ANNUAL REPORT OF THE COMMITTEE ON INDEXING CHEMICAL LITERATURE.*

THE Committee on Indexing Chemical Literature presents to the Chemical Section its Fifteenth Annual Report, covering the twelve months ending August, 1897.

Works Published.

"Re-calculation of the Atomic Weights." By Frank Wigglesworth Clarke. New edition, revised and enlarged. Constants of Nature, Part V. Smithsonian Miscellaneous Collections, 1075. City of Washington, 1897. Pp. vi.—370. 8vo.

"Index to the Literature of the Periodic Law." In: "Development of the Periodic Law." By F. P. Venable. Easton, Pa., 1896. 12mo.

"Partial Bibliography of Argon." By C. Le Roy Parker. Accompanying his paper: "Our Present Knowledge of Argon." *J. Am. Chem. Soc.*, xix., 124 (Feb., 1897).

"Bibliography of Agricultural Chemistry (American)." Bulletins of the Office of Experiment Stations, United States Department of Agriculture.

* In our Fourteenth Annual Report the following correction should be made: for Bulletin No. 9 read Bulletin No. 19, and add Bulletin No. 27 (1896).

A card-index to Experiment-Station Literature is issued by the Office of Experiment Stations; this is sent *gratis* to all the Agricultural Colleges and Experiment Stations in the United States, and is sold to a limited number of

individuals and institutions. Eleven thousand cards had been distributed prior to September, 1896.

The detailed index included in each volume of the *Experiment Station Record* contains numerous references to chemical articles published by the Experiment Stations in the United States and in foreign countries.

"Abstracts of Chemical Work in Agricultural Science," published in: *Experiment Station Record* issued by the Office of Experiment Stations, United States Department of Agriculture.

These Abstracts were begun in vol. i., No. 1 (September, 1889). Abstracts of foreign investigations were added beginning with vol. ii., No. 8 (March, 1891), and these have been included, with a quite rapid growth in the field covered, up to the present time. The work is in charge of Dr. E. W. Allen, who is assisted by Mr. W. H. Beal in the departments of fertilisers and soils, and by the Committee on Abstracting of the Association of Official Agricultural Chemists.

"The Review of American Chemical Research," edited by Prof. Arthur A. Noyes, began in April, 1895, and formerly published in the *Technology Quarterly*, is continued in the *Journal of the American Chemical Society*.

"Periodicals relating to Chemistry and Physics" in the New York Public Library and Columbia University Library. Bulletin of the New York Public Library, Astor, Lenox, and Tilden Foundations. Vol. i., No. 6. June, 1897. Page 152.

A very convenient check-list compiled with bibliographical accuracy, especially useful to students residing in New York and vicinity.

"Bibliography of the Analysis of Chrome-iron Ore, Ferro-chromium, and Chrome-steel." By S. Rideal and S. Rosenblum. *CHEM. NEWS*, vol. lxxiii., p. 2. (Jan. 3, 1896).

"A Bibliography of the Chemistry of Chlorophyll," by L. Marchlewski, accompanies his monograph: "Die Chemie des Chlorophylls." Hamburg and Leipzig. 1895. 8vo.

Reports of Progress.

"A Bibliography of the Metals of the Platinum Group," 1748—1896, by Professor James Lewis Howe, has been completed, and after examination by your Committee has been recommended to the Smithsonian Institution for publication. The work is now going through the press.

"A Review and Bibliography of Metallic Carbides," by Mr. J. A. Mathews, of Columbia University, was submitted to your Committee, and, after examination by each member, the MS. was returned to the author for minor improvements. The suggestions of the Committee were promptly accepted by Mr. Mathews, and the revised work has been recommended to the Smithsonian Institution for publication.

"A Bibliography of Basic Slags, Technical, Analytical, and Agricultural," has been completed by Karl T. McElroy, of the Division of Chemistry, U.S. Department of Agriculture. The channel of publication has not been determined.

The second edition of the "Catalogue of Scientific and Technical Periodicals," 1665—1895, by Dr. H. Carrington Bolton, is entirely printed, but publication is deferred owing to the preparation of a new Library Check List, with which it will be accompanied. The new edition contains 8603 titles.

"A Supplement to the Select Bibliography of Chemistry," 1492—1896, has been completed by Dr. H. Carrington Bolton, who has presented the MS. to the Smithsonian Institution. This Supplement contains about 9000 titles, including many chemical dissertations, and is brought down to the end of the year 1896.

* Advance proofs from the *Proceedings of the American Association for the Advancement of Science*, vol. xlv., 1897. Communicated by H. Carrington Bolton.

Dr. C. H. Jôuet reports his "Index to the Literature of Thorium" nearly finished.

Dr. F. W. Traphagen reports "fair progress" on his "Index to the Literature of Tantalum."

Mr. George Wagner reports that he has made progress on the "Bibliography of Oxygen."

Mr. H. E. Brown, under the direction of Professor A. B. Prescott, is preparing a "Bibliography of the Constitution of Morphine and Related Alkaloids."

Professor William Ripley Nichols, of the Massachusetts Institute of Technology, at the time of his death left an unfinished "Index to the Literature of Carbonic Oxide"; the MS. was taken in hand by Professor Augustus H. Gill, of the same institution, who has done considerable work upon it; he now reports that he is not in a position to finish the task, and he is perfectly willing to relinquish the large amount of material accumulated to anyone who would undertake to complete it.

Professor Clement W. Andrews, formerly of the Massachusetts Institute of Technology, and now Librarian of the John Crerar Library, Chicago, reports that he is obliged to abandon work on his "Index to the Literature of Milk," and will be very glad to turn over the material to anyone who cares to undertake to complete the bibliography.

It has always been the aim of the Committee on Indexing Chemical Literature to prevent duplication of work, but failure to inform the Committee of work in progress may defeat this undertaking. An announcement in the Fourteenth Annual Report, of certain work having been nearly completed, surprised a chemist in another part of the country, and has led to the abandonment by the latter of much laborious indexing.

In conclusion, the Committee repeats the statement that it labours to encourage individual enterprise in chemical bibliography, and to record in the annual reports works issued and works in progress.

Address correspondence to the Chairman, at Cosmos Club, Washington, D.C.

Committee:—

H. CARRINGTON BOLTON, Chairman,
F. W. CLARKE,
A. R. LEEDS,
A. B. PRESCOTT,
ALFRED TUCKERMAN,
H. W. WILEY.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, June 17th, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

(Concluded from p. 70).

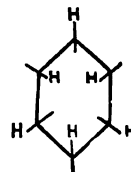
*80. "On a Space Formula for Benzene." By J. NORMAN COLLIE, Ph.D., F.R.S.

In this formula six tetrahedra (to represent the six carbon atoms) are arranged symmetrically in space equidistantly from a common centre, and so that they would occupy the six solid angles of an octahedron. They are connected also symmetrically with one another by single linkings. If the six hydrogen atoms (of benzene) are then arranged symmetrically on these tetrahedra, it will be found that there will be three on one side of the figure and three on the other side.

Movement in this arrangement might take place in two ways; a movement of each tetrahedron about its own

centre, and a movement of each tetrahedron about the centre of gravity of the whole mass.

In the first case, simultaneous rotational movement of each tetrahedron about its centre would bring the combined hydrogen atoms towards the centre of the mass in two distinct sets; those on the 1, 3, 5 carbon atoms and those on the 2, 4, 6 carbon atoms, and a projection of this configuration might be expressed as follows:—



In the second case, movement about the common centre would alter the relative positions of the tetrahedra with regard to one another, bringing into play the six unsaturated points of attraction on these tetrahedra. The projection of these different phases can be represented by the formula given in Fig. 1.

This space formula is therefore in complete accord with that of Kekulé and the centric formula, and shows how they are mutually convertible the one into the other. It also shows how the supposed double bindings in the Kekulé formula shift between the carbon atoms, thus rendering two orthochlorobenzenes impossible. But it differs from both, in that it shows how there may be two distinct sets of hydrogen atoms, and that when one set is inside the molecule, the other set is outside the molecule.

It can offer an explanation also of the fact that when one set of groups is present in a benzenoid compound, further substitution gives ortho- and para-derivatives; whilst, when another set is present, on further substitution meta-derivatives only are formed.

When chlorine adds on nitrobenzene, the chief product is metachloronitrobenzene. The nitro-group belongs to the group that favours the production of meta-derivatives. This nitro-group, being in a sense unsaturated, might possess a certain amount of "residual affinity" which would be sufficient to attract the entering chlorine molecule, and direct it towards the hydrogen atoms that come to the centre at the same moment that it does itself. (See Fig. 2).

On the other hand, in the case of chlorobenzene, if nitric acid be allowed to react with it, no such additive compound would be produced, and the attraction of the three hydrogen atoms attached to the other three carbon atoms might just be sufficient to determine its reaction with them. (See Fig. 3).

DISCUSSION.

Professor TILDEN thought the paper a valuable contribution to the theory of the construction of the benzene molecule, though he felt doubtful about the validity of the division of the substituents into saturated and unsaturated, since this distinction was, in any case, not a sharp one.

Mr. SWORN considered that the formula proposed did not differ materially from the octahedral formula of Victor Meyer and others, nor did the explanation by the law of substitution now suggested differ substantially from one proposed by himself in a paper published in the *Philosophical Magazine*.

Dr. KIPPING said he was not satisfied with the conclusion that three hydrogen atoms in benzene occupy different positions to the other three. If this were true then two monosubstitution derivatives become possible. From the readiness with which rings are formed from side groups, it would appear that the meta-position corresponds with the ortho-position.

Mr. FRISWELL said that the ordinary nitration of toluene gave a mixture of about 65 parts of orthonitrotoluene and 35 of paranitrotoluene, or very nearly 2 to 1. In conjunction with Dr. T. A. Lawson he had endeavoured

FIG. 1.

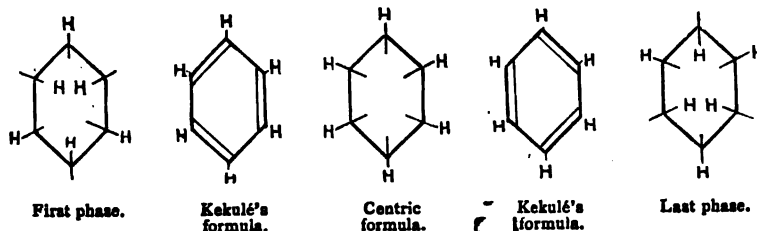


FIG. 2.

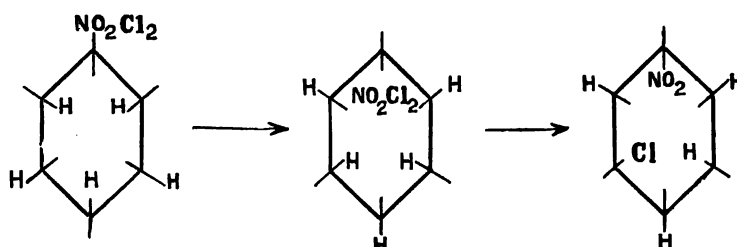
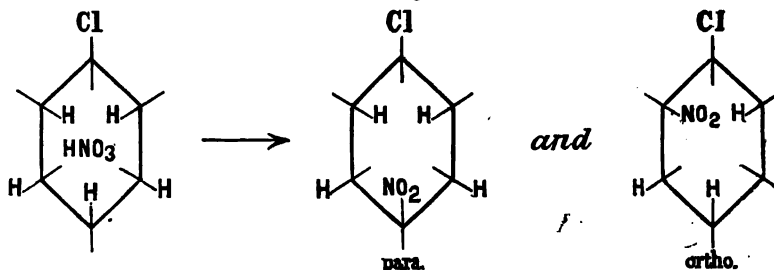


FIG. 3.



to alter these proportions. Every possible variation of temperature down to nitration at or near zero or as high as 40° , every variation of nitrating mixture from large proportions of sulphuric acid to the use of nitric acid alone, every variation in the order and rate of mixture, the nitration of toluene in which paranitrotoluene had been previously dissolved, had been tried. Yet no important variation of the proportions of the two products had been produced. Mr. A. G. Green had repeated these experiments, and confirmed them. The space formula now suggested by Dr. Collie afforded an explanation, since there were two orthohydrogen atoms during the rotation postulated inside the ring, and only one para-hydrogen atom in that condition. He had long considered this problem, and he had, in conjunction with Mr. C. Mills, commenced to experiment in order to ascertain whether the results might not be due to the existence of two isomeric toluenes.

Professor COLLIE, in reply, pointed out that the space formula for benzene which he had proposed was necessarily similar in many respects to others, notably those of Vaubel and Sachse; but the point on which he wished to lay especial stress was, that there were two sets of hydrogen atoms, and that when one set was inside the molecule the other set was outside.

90. "Note on the Formation of Diacetanilide." By GEORGE YOUNG, Ph D.

The introduction of a second acetyl group into acetanilide has been described in recent years by several authors. Kay (*Ber.*, 1893, xxvi., 2853) treated acetanilide with acetyl chloride at $170-180^{\circ}$ from three to four hours. Bistrzycki and Ulfers (*Ber.*, 1894, xxvii., 91)

heated a mixture of acetanilide and acetic anhydride, under pressure, eight to ten hours at $200-205^{\circ}$. Blacher (*Ber.*, 1895, xxviii., 2356) boiled sodio-acetanilide suspended in xylene with acetic anhydride. Tassinari (*Gazz.*, 1894, xxiv., i., 61) acted with acetyl chloride on sodio-acetanilide suspended in benzene. The English and German abstracts of Tassinari's paper state that this author also prepared diacetanilide by treatment of acetanilide with acetic anhydride, but the following quotations from the original paper show that the method used consisted in boiling a mixture of acetanilide, acetic anhydride, and sodium acetate on a reflux apparatus for some hours. In the introduction to his paper, Tassinari makes the general statement, "le diacidanilidi si formano anche con anidride acetica ed acetato sodico a ricadere." It is true that in describing the preparation of diacetanilide, he does not mention sodium acetate—"Trattando dell' acetanilide con anidride acetica, come è detto sopra per la formilide . . ." but the passage referred to runs: "Scaldando a ricadere per alcune ore un misto di formilide, anidride acetica, ed acetato sodico. . . ." Further, in a later paper (*Gazz.*, 1894, xxiv., i., 444), in which the work of Bistrzycki and Ulfers is quoted, no notice is taken of a statement by these authors (*loc. cit.*) that, although acetanilide undergoes some change when boiled for two hours with acetic anhydride, they were unable to obtain any pure product from the reaction.

The introduction of the second acetyl group takes place much more easily than might be imagined from the results quoted. If acetanilide be boiled with two to three times its weight of acetic anhydride for half an hour, over 75 per cent is converted into the diacetanilide, which may

be easily purified by the following method:—The cooled product is shaken with benzene and sodium carbonate solution. After drying over calcium chloride, the benzene solution is distilled as far as possible on the water-bath, and the residue treated with light petroleum. The unchanged acetanilide is removed by filtration, and the filtrate evaporated. The residue solidifies on cooling to a crystalline mass, melting at 37–38°. A single recrystallisation, by extraction with cold light petroleum and evaporation of the extract, is sufficient to purify it for analysis, when it melts sharply at 38°.

0.1792 gave 0.4449 CO₂ and 0.1025 H₂O. C=67.71; H=6.35 per cent.

C₆H₅N(COCH₃)₂ requires C=67.79; H=6.21 per cent.

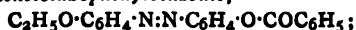
Part of the diacetanilide formed is probably hydrolysed by the treatment with sodium carbonate solution, but by working with not too large quantities, and performing the purification as rapidly as possible, a yield equal to the weight of acetanilide taken may easily be obtained.

91. "Derivatives of Phenetol Azo-phenols. By J. T. HEWITT, M.A., D.Sc., Ph.D., T. S. MOORE, and A. E. FITT.

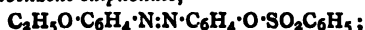
One of the authors has shown (*Ber.*, 1895, xxviii., 799) that certain substitution derivatives of benzeneazophenol can form addition products with half a molecule of water, differing very sharply in colour and other physical properties from the corresponding anhydrous compounds. In order to obtain further knowledge of this subject, various series of substituted benzeneazophenols are being prepared and examined. This communication deals with ortho- and para-phenol azophenols, C₂H₅O·C₆H₄·N:N·C₆H₄·OH, the examination of the meta-derivative being deferred. When a paraoxyazo-compound does form an addition product with water, the addition of the latter can be most frequently brought about by dissolving the azo-compound in benzene and precipitating it by gaseous hydrogen chloride as a hydrochloride of the general formula, X·N:N·C₆H₄·OH, HCl, and decomposing this with water. Frequently the molecule of hydrogen chloride is thus replaced by a half molecule of water.

Orthophenetolazophenol was prepared according to the method given by Jacobson and F. Meyer (*Annalen*, 1895, cclxxxvii., 213). The melting-point was found to be 128° C. (corr.). Jacobson gives 131° C. The *hydrochloride* melted between 124° and 129° C. On decomposition with water, the azophenol was regenerated; after air drying it melted at 127–128°. It may be assumed that no water had been added. In order to further characterise the azophenol, the two following derivatives were prepared.

Orthophenetolazophenylbenzoate,—



light scarlet needles, m. p. 98° (corr.). *Orthophenetolazophenylbenzene sulphonate*,—



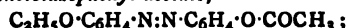
brilliant red needles, m. p. 83° (corr.).

Paraphenetolazophenol has been prepared by Riedel (*D. R. P.*, xlviii., 453), and also by Jacobson and F. Meyer (*Ann.*, 1895, cclxxxvii., 215). The former gives the melting-point as 104.5° C., the latter as 125–126°. The method of the latter chemists was used to prepare the compound. The melting-point was found to be 125° (corr.). The *hydrochloride* gave no very sharp melting-point, beginning to melt at 131°, fusion not being complete until 154°. On treatment with water, a pale yellow powder is obtained melting at about 100°. The same substance may also be obtained by dissolving phenetolazophenol in glacial acetic acid, adding fuming hydrochloric acid, and pouring into water. After careful drying in air the substance melted at 104–109° C. Apparently this substance consists of equimolecular proportions of water and paraphenetolazophenol.

	Calc.	C ₁₄ H ₁₄ N ₂ O ₂ ·H ₂ O.	Found.	Mean.		
C	..	64.62	65.10	64.49	64.39	64.68
H	..	6.15	6.40	6.29	5.95	6.21

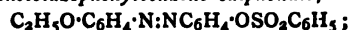
The following derivatives of paraphenetolazophenol have been prepared.

Paraphenetolazophenyl acetate,—



yellow leaflets, m. p. 118° (corr.). *Paraphenetolazophenyl benzoate*, C₂H₅O·C₆H₄·N:N·C₆H₄·O·COC₆H₅; small reddish brown crystals, m. p. 126° (corr.).

Paraphenetolazophenylbenzene sulphonate,—



large pale yellow plates, m. p. 104° (corr.).

92. "δ-Ketopinic Acid and Camphoic Acid." By W. S. GILLES and F. F. RENWICK.

In the description first given of ketopinic acid (*cf.* Armstrong, *Trans.*, 1896, lxix., 1397), it was stated that the acid was optically inactive, even when prepared by oxidising the most active chlorocamphydrene (pinene hydrichloride) obtainable. As it was a matter of importance to determine whether the inactivity was an inherent property or due to compensation, the authors have applied Pasteur's method, and have succeeded in separating a dextrorotatory modification by fractionally crystallising the mixture of salts obtained by combining the inactive acid with strychnine. δ-Ketopinic acid has the same melting-point as the "inactive" acid from which it is separated.

In their previous note (*Proc.*, 1897, xiv., 64) the authors have stated that when ketopinic acid is oxidised by permanganate, it is converted into a tribasic acid resembling the camphoic acid described by Marsh and Gardner; they are now able to state that the product is camphoic acid, having obtained from it the *cis*- and *trans*-camphopyric acids and camphopyric anhydride of these chemists.

An amount of camphoic acid equal to 80 per cent of the weight of the acid oxidised may be obtained by boiling ketopinic acid with a solution containing 50 per cent of nitric acid and adding small quantities of stronger acid (*d*=1.42) from time to time, as the action proceeds.

The authors will endeavour to ascertain if pinophanic and camphoic acids also exist in optically active forms, and what is the behaviour of active ketopinic acid on oxidation. Acids which are probably *cis*- and *trans*-forms of pinophanic acid have already been obtained.

93. "Note on Stereoisomeric Di-derivatives of Camphor, and on Nitrocamphor." By T. M. LOWRY, B.Sc.

Having learned from Dr. Armstrong that, in the course of his early studies of camphor derivatives, he had observed that the substances obtained on the one hand by chlorinating bromocamphor and on the other by brominating chlorocamphor are apparently different, the author has submitted the two products to examination.

Brominated chlorocamphor, according to Cazeneuve, melts at 51.5°. The author finds that, on warming a mixture of chlorocamphor and bromine and once crystallising the product from spirit, well-defined crystals are obtained which melt at 53–55°; on analysis, these give results showing them to be bromo-chlorocamphor. By repeated re-crystallisations from a variety of solvents, this product, however, may be resolved into two fractions, alike in composition, but widely different in specific rotatory power. The less soluble product, after being twenty-five times re-crystallised, fused at 61°; its specific rotatory power was [α]_D = 16°. The more soluble fraction—obtained by evaporating the mother liquor, distilling the residue with steam, and re-crystallising the product from dilute spirit—fused at 55°; its specific rotatory power was [α] = 63.9°.

On directly chlorinating bromocamphor, an oil was obtained which could not be caused to crystallise; but a crystalline chlorinated bromocamphor was obtained without difficulty by heating bromocamphor with sulphuryl chloride at 130°. After being twice crystallised from spirit, the product fused at 56°; its specific rotatory power was [α]_D = 25.7° instead of 51°—the value observed in the case of the corresponding product from chlorocamphor. As in the

With a small quantity of benzene hydrochloride, C_6H_7Cl , was also obtained by the action of alcoholic potash on trichlorhexanaphthene. It boiled at $135-140^\circ$, and an analysis gave the following result:—C, 63.20; H, 5.91; Cl, 30.36. Calculated for C_6H_7Cl :—C, 62.93; H, 6.12; Cl, 30.95. Hexanaphthene, when heated with fuming nitric acid, was found to be oxidised to adipic acid, as stated by Markownikoff (*Ber.*, 1897, xxx., 975). As both this chemist and Zelinsky appear to have obtained methyl pentamethylene by the action of hydriodic acid on derivatives of hexanaphthene (*Ber.*, 1897, xxx., 387, 1214), it became of interest to see whether the hydrocarbon itself

would undergo isomeric change under similar conditions. Hexanaphthene boiling at $80.0-80.1^{\circ}$ was therefore heated in a sealed tube with about five times its volume of hydriodic acid, sp. gr. 1.96, and a little amorphous phosphorus. The tube was heated to about 160° for six hours, from 250° to 270° for three hours, and was maintained at about 250° for four hours longer. The hydrocarbon, after being washed and dried, boiled almost constantly at 80° , showing that it was unchanged hexamethylene.

Zelinsky has found that methylhexamethylene is converted into dimethylpentamethylene by heating with hydriodic acid (*Ber.*, 1897, xxx., 1532); it is therefore interesting to note that hexamethylene itself appears to be much more stable than its derivatives.

It is hoped to obtain a fresh supply of the substance by the fractional distillation of Galician petroleum, when the experiments will be continued.

stearine, which latter substance reduces the melting-point of the mixture below that of either of the ingredients.

We regret to find the un-English orthography "vise" for "vice" adopted in the chapter on the purification of crude oil.

A very important feature of this work is the catalogue of patents and list of patentees. Perhaps we may be permitted to regret the number of patents which form one of the difficulties of the shale-oil trade.

The Analysis of Food and Drugs. Part I.—Milk and Milk-products. By T. H. PEARMAIN and C. G. MOOR, M.A. (Cantab.). Pp. 132. London: Baillière, Tindall, and Cox. 1897.

As this work may run to some 800 or 900 pages, it has been decided to bring it out in several parts, so that each portion may be as complete as possible and contain all the latest information. Each part will be complete in itself, and will have its own index, while a general index will be issued with the last part.

The chief need at the present time is the formation of standards to which the various articles of food should conform, especially in the case of milk, one of the most important and universal foods we have.

It is to the analysis and examination of milk and milk-products that this part is devoted. Milk, which has been described by a well-known doctor as a solid food, is the only article occurring in Nature which combines in the right proportions all the necessary elements requisite to secure proper nutrition, but it is too voluminous to serve as the sole food of adults: it is to prevent this proportion being upset by the interference of the milk-seller that has brought milk analysis into the prominent position it now holds, and renders necessary the small army of inspectors who are constantly on the look-out for adulteration.

For our knowledge of the composition of milk we are indebted, in this country, mainly to the elaborate and prolonged researches of Adams, Bell, Hehner, Stokes, and others. It is found that genuine cow's milk does not normally differ much from the typical analysis here given; it is on the rarest occasions that there is less than 3.5 per cent of fat present, while the average is 4 per cent, and the solids-not-fat may be taken as 8.5 per cent.

It is not surprising that the adulteration of milk to a certain degree is so universal, when in most cases 20 per cent of water, or 30 per cent of skimmed milk, may be added without the resulting mixture falling below the present standard.

The comparison of many thousands of samples a year, for the six years 1890-5, shows a regular steady rate of adulteration of about 12 per cent, and the authors consider that "it is not too much to say that the disgraceful state of the milk trade in this country is fostered, if not actually caused," by the ridiculously low standard adopted by the Laboratory of the Inland Revenue Department, "by which, of course, analysts are compelled to abide, or take the risk of being over-ruled by the referees, who are thought to be infallible in the eyes of some magistrates."

There are several different methods of determining the fat, such as the mechanical methods, and simple extraction with a solvent of the dried milk by Bell's, Adams's, or Macfarlane's method; or extraction of the fat by ether, after destroying the casein by acid. All these methods, as well as those for the estimation of proteids, are fully described in these pages.

The most commonly used preservatives to be found in milk are borax, formaldehyd, salicylic acid, and potassium chromate, and there is no doubt that formaldehyd, or formalin,—by which name it is also known,—being, it is alleged, the most effective, will, when it becomes more generally known, supersede boric acid as a preservative. Two or three drops of formalin in a pint of milk are said to keep it fresh for several days, and the addition of 0.05 per cent should preserve milk for months.

NOTICES OF BOOKS.

A Practical Treatise on Mineral Oils and their By-products; including a Short History of the Scotch Shale Oil Industry, the Geological and Geographical Distribution of Scotch Shales, Recovery of Acid and Soda used in Oil-refining, and a List of Patents relative to Apparatus and Processes for obtaining and refining Mineral Oils. By ILTYD I. REDWOOD, Member of the Society of Chemical Industry (England). London: E. and F. N. Spon, Lim., 125, Strand. New York: Spon and Chamberlain. 1897.

MR. ILTYD I. REDWOOD merits the gratitude of the chemico-technical world for his comprehensive work, the value of which is enhanced by the portrait of the late James Young, the founder of the Scottish mineral oil industry.

The first chapter treats of the history of the trade. James Young was what is called a "self-made man." He became demonstrator and assistant to Professor Graham, and amidst many difficulties laid the foundation of the new manufacture. The famous Bathgate Oil-works were erected in 1851, and after many ups and downs have reached their present position.

Mr. Redwood foretells that unless a unanimous and harmonious combination of the Companies soon takes place, we "shall in a few years be left to mourn the loss of one of Scotland's most important industries."

The true shales are confined to a district extending 12 miles north and south by about 25 miles east and west. They lie in the calciferous sandstone series, lying between the Mountain and the Buriehouse limestones.

Shale is distinguished from coal by the lack of the intense blackness of the latter mineral; it is less easily broken, and then displays a conchoidal fracture.

The proportions of crude oil yielded by the mineral vary in different localities. Thus the Denbrae shale yields 10.90 gallons of crude oil per ton, whilst the true Boghead mineral yields from 85 to 128 gallons and the methyl brown coal from 65 to 90 gallons. A large yield of crude oil is not always a proof of high value, since a high yield of oil is often accompanied by a low percentage of paraffin wax and an abundance of lubricating oils of low specific gravity.

We learn that mineral oils alike in specific gravity often vary greatly in viscosity. Hence consumers prefer to buy oils by the viscosity test rather than by specific gravity. Still it is not safe to rely exclusively upon viscosity. The consumer is therefore recommended to have the cold test and the viscosity of samples both carefully ascertained. The relation between these two sets of data is shown in a table given on p. 221. Further tables (p. 225) show the melting-points of mixtures of wax and

Potassium chromate is not known as a milk preservative in this country, but it is stated to be used on the Continent.

Milk contains, as a rule, a large number of bacteria, for the most part derived from the external conditions and surroundings of the cow; the numbers may even run so high as three or more millions per c.c.; and as many of these, which are capable of causing disease, do so by producing toxic decomposition products, which is greatly increased by a rise of temperature, it is essential that the very greatest care should be taken to keep the temperature as low as possible, from the time when the milk is drawn to the time when it is consumed or cooked. There is little doubt that the great mortality among young children from intestinal tuberculosis, is due to the presence of the tubercle bacillus in the milk used.

The sterilisation of milk is a most important subject, and we are sorry to see that it is very lightly passed over in this volume; it is of course very necessary to have everything connected with the milk supply scrupulously clean, but it is also necessary to have some regular recognised process by which the ubiquitous *Bacillus tuberculosis* can be killed instead of remaining to breed in the milk. Pasteurisation is coming widely into use on the Continent, and we should be glad to hear of its general adoption in this country.

The authors are to be congratulated on having produced a most useful and readable book, and we can only hope the parts yet to come will be worthy of Part I.

Monopolies by Patents, and the Statutable Remedies available to the Public. By J. W. GORDON, of the Middle Temple, Barrister-at-Law. Pp. 300. London: Stevens and Sons, Lim. 1897.

In his Preface the author explains his reason for taking the word "Monopoly" for the purpose of the title of this book. The word Monopoly, as now generally accepted, no longer has the meaning in law that it had in the time of Blackburn; want of a better one is the reason of the present title used in the now-forgotten sense which it formerly had, a sense in which it implies that not a single individual must be prevented from freedom to follow any lawful trade.

The Patent Law took its rise from the time of Queen Elizabeth, when, owing to the interference caused to lawful trade by the large number of monopolies she had granted, a reaction was produced in the public mind, which might have resulted in a political convulsion had she not afforded some relief by recalling a few of the most iniquitous of the monopolies she had granted.

The law, which has since become the law of England and the Colonies, besides that of many foreign States, was laid down in the year 1602. It was some years, however, before popular feeling was satisfied with the action taken by King James II. One circumstance, however, which helped to set the public mind at rest was the publication of the King's Book of Bounty, a volume remarkable for its career, and which is reprinted in *facsimile* as a part of this work in Appendix I.

In 1621 the grievance had again assumed serious proportions, and an Act was passed actually comprising a declaration the King had made some years previously in a moment of candour. The fourth section of this Act was considered of great importance in protecting the public against monopolies; nothing is omitted which could make proceedings easy for an aggrieved person in assertion of his rights; in fact, it might be considered as the utmost Parliament could do to protect the weak against the strong. The growing tendency in our own times of patentees to threaten legal proceedings against all and sundry, has given patents a new and formidable character. The interests of the public as against that of the patentee is a matter of great and growing importance, and in which vast interests are involved.

The power to seize infringing goods is no longer included in a patent grant, but it is at the present day a common form of pleading to ask for the giving up of such goods, as part of the damages asked for, and the Courts do not shrink from allowing this in certain cases: it is a recent innovation, dating from the year 1846, and the author considers that it arises from what are, it is contended, insufficient authorities. The earliest report in which the author has been able to find an order for destruction is in 1864, but there seems to have been some confusion in its drawing up. But in *Tangye v. Stott*, in 1866, the defendant had the option of giving up or destroying the incriminating goods, so that it does not appear to be definitely recognised that the property in them may pass to the patentee.

The celebrated Case of Monopolies, which the author considers to be one of the most interesting reports in the English Law Books, is here, and now for the first time collected and published in complete form. Considered altogether, the author thinks "it is not too much to say that in the whole of legal history there is no other deliverance in any tongue which has proved to be so fruitful of results, nor any which has contributed more to the advancement of society in modern times."

The book cannot fail to be of great interest to those whose minds are cast in a legal mould, and it will undoubtedly be of great service to those whose business comes within its scope. Besides an excellent general Index, there are 19 pages of Cases which are referred to in the work, also carefully indexed.

CORRESPONDENCE.

RELATIONS BETWEEN MELTING-POINTS AND LATENT HEATS OF FUSION.

To the Editor of the Chemical News.

SIR,—In the *CHEMICAL NEWS* of June 11 (vol. lxxv., p. 278) there appeared a paper by Dr. Joseph W. Richards dealing with relations existing between the absolute melting-points (T) and the latent heats of fusion (L) of the metals. Dr. Richards, in 1893, announced that the latent heats of the metals bears a simple ratio to the heat required to raise the metal from the absolute zero to its melting-point.

In June, 1895, in a short paper I forwarded to the Chemical Society, I proposed this relation, which is practically the same as that of Dr. Richards:—"That where the valency is the same, the absolute temperature,

$$T, \text{ of the melting-point is proportional to the quantity } \frac{L}{S};$$

L being the latent heat of fusion, and S being the specific heat which has to be treated as a constant. The quantity $\frac{L}{S}$ I termed the temperature equivalent of fusion. Expressed in this way the numerical agreement was very striking.

Dr. Richards does not include the non-metals in expressing his relation; but between bromine and iodine there is an exact agreement, although the number obtained from these elements does not tally with that given by sodium, potassium, and silver—metallic monovalent elements—but is almost exactly the number given by the latter.

This relation of mine was proposed as a corollary to a relation brought forward by Mr. Holland Crompton before the Chemical Society, in April or May, 1895, when he proposed the relation
$$\frac{L}{AT} = KV; \text{ A}$$

being the atomic weight, V the valency, and K a constant. Previous to this (in May or June, 1895), I had read a

paper before the Physical Society, in which I brought forward this relation,—

$$\left(T + \frac{L}{S}\right)a = \text{constant},$$

as holding between the members of any family in the periodic classification of the elements; a being the mean coefficient of expansion between T and -273° . The values thus found were satisfactory, but the argument following which the relation was obtained was received with considerable adverse comment. This relation, combined with that of Mr. Holland Crompton, mentioned above, leads to these relations, with the same restriction as before:—

$$Ta = \text{constant}.$$

$$\frac{L}{S}a = \text{constant}.$$

The calculated values of these quantities were confirmation of the truth of the relations.

At that time I had not seen Piçet's rule,—

$$Ta\sqrt[3]{V} = \text{constant};$$

V being the atomic volume of the element in question. Now, if $Ta = \text{constant}$ for the members of a periodic family, and also $Ta\sqrt[3]{V} = \text{constant}$ generally, then must $\sqrt[3]{V}$ be constant for the members of a periodic family.

Dr. Richards concludes his paper by showing that, by combining his relation with Piçet's rule, the relation $L = 2.1T = \frac{9.5}{a\sqrt[3]{V}}$ is obtained. On exactly the same

grounds, by combining my relation $Ta = \text{constant}$ with Piçet's rule, we obtain the relation $L = 2.1T = \frac{\text{constant}}{a}$.

This relation, as before, being restricted to members of the same periodic family.

Dr. Richards, in his paper, concludes with calculating certain unknown latent heats. For one of these (thallium) he gives the value 5.8; this has been determined by Messrs. Neville and Heycock—unless my memory misleads me—to be 5.1.

Dr. Richards quotes the latent heat of copper as 43; this quantity was not available when I read my paper before the Physical Society; I stated then that a value of about 40 would bring that element into line with its fellow members in its periodic relationships.

I regret that I am unable to quote any actual figures, but placed as I am—on a sugar estate some distance from a town—no data are available for me.

A short abstract of my paper appeared in the *CHEMICAL NEWS* (vol. lxxi., p. 303). The Physical Society did not think fit to publish it in their *Proceedings*.

In conclusion, I wish to say that what I have written is in a friendly spirit, and that I have no desire to break a lance with so redoubtable an antagonist as Dr. Richards.—I am, &c.,

NOËL DEER.

The Laboratory,
Plantation, Windsor Forest,
West Coast, Demerara.

On Cafetannic Acid.—P. Cazeneuve and M. Haddon.—Recent researches based on the action of phenylhydrazine, either on cafetannic itself or on the sugar formed by its splitting up, show us that the formula adopted for cafetannic acid is entirely wrong, and that the sugar formed is a new sugar, which cannot be confounded with mannitan or with any known sugar.—*Journal de Pharmacie et Chimie*, vi., No. 2.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Journal de Pharmacie et Chimie.

Series 6, vol. v., No. 12.

Action of Sodium on Albumenoid Matters.—E. Lépine.—The halogen proteids of Blum and the iodised albumen of Renault are very complex and variable in their composition and their action on organisms. The author has therefore directed his endeavours to producing a compound always identical and whose composition is well known: for this purpose he used an aqueous solution of iodine, and let it act in the cold, so as to avoid violent reactions. He has worked on casein as well as albumen. The experiments on pure casein were unsatisfactory owing to its feeble solubility, so he had recourse to milk itself, and to this he added a solution of iodised iodide (*iodo ioduré*) until a slight excess could be detected by means of chloroform, even after standing twenty-four hours. At the end of this time an equal volume of distilled water, containing a little acetic acid, is added; there is thus obtained a yellowish brown coagulum, which is well washed with water and treated with weak soda: the colour disappears, and a soluble caseinate is formed. From the filtered liquid we can again precipitate the casein by acetic acid. To obtain it perfectly pure this must be repeated five or six times; it then has a constant composition and leaves no ash on calcination. There is no doubt that this body contains iodine in its molecule, for if treated with soda and nitrate of soda, and taking up the mass with water, it is easy to detect the presence of iodine. We may therefore conclude that this casein is actually iodised. To show its constant composition five different analyses were made, and they gave average results of 21.6 per cent iodine and 14.15 per cent nitrogen, the greatest variations being 20.0 and 22.4 per cent for iodine and 14.1 and 14.23 per cent for nitrogen. The first yellowish brown coagulum loses its colour not only when treated with soda, but also by losing a portion of its iodine when treated with hyposulphite of soda or sulphurous acid; but in neither case is all the iodine removed. The author hopes that, owing to its constant proportion of iodine, this iodo-casein may become useful in therapeutics, and he proposes carrying out some experiments on digestion with men and animals.

Presence of Lead in some Sterilised Artificial Serums.—M. Chevreton.—A case of poisoning by an injection of an artificial serum having been observed, M. Chevreton sought for the cause, and he found that, by keeping an artificial physiological serum (7 per 1000 solution of NaCl) at 120° for twenty minutes, in different flasks, when lead glass was used the liquid became charged with this poison; and if this serum be used for subcutaneous or intra-venous injections, there is naturally a very grave risk of lead-poisoning: the presence of lead is easily proved by means of iodide of potassium.

Series 6, vol. vi., No. 1.

On a Method of Oxidation and Chlorination.—A. Villiers.—The addition of a trace of a salt of manganese will, in many cases, accelerate oxidation. This action can be explained by the production of easily decomposable salts, similar to those produced in the preparation of chlorine; the presence of traces of manganese can not only keep the oxidation in media containing special oxidising materials, but can in certain cases facilitate the direct absorption of oxygen from the air. The conditions under which oxidation takes place, under the influence of salts of manganese, in the case of oxalic acid, are rather curious. The disengagement of gas can be seen in the

cold. It will last several weeks, probably even several years. These reactions, of which several are already known, have the characteristics of fermentations produced by chemical ferments, and may be rightly classed as *mineral ferments*.

On Isomerism of Pilocarpidine and Pilocarpine.—A. Petit and M. Polonovski. — Two experiments have sufficed to satisfy the authors that the transformation of pilocarpine into pilocarpidine takes place inter-molecularly. After boiling a salt of pilocarpine with an excess of dilute soda for some hours, in a flask fitted with a vertical condenser, the condenser was lowered and three-quarters of the solution distilled, although nearly all the pilocarpine was transformed, they were unable to detect the least trace of methylic alcohol; *there is therefore no elimination of a methyl group* during the transformation of pilocarpine into pilocarpidine. Another experiment was still more decisive. Chlorhydrate of pilocarpine, kept in a state of fusion for a few instants, was entirely converted into chlorhydrate of pilocarpidine. Full details, weights, and temperatures, employed in this experiment, are given.

Contribution to the Study of the Preparation of Ordinary Ether.—L. Prunier. — In making ether from sulphuric acid and alcohol we are in the habit of regarding the transformation as in two successive phases, forming a complete cycle, being indefinitely renewed; this continuity does not exist in practice. The reaction certainly does take place in an acid medium, but the presence of sulphuric acid is not indispensable; etherification takes place in its absence, the acidity being due to sulphurous acid, sulphovinic acid, or its derivatives, the latter perhaps being of considerable importance. Isoethioniac acid and its derivatives are taken as a type of the group, as their properties are well known, and in the author's experience the sulphonic derivatives have been characterised by groups and not by distinct species. Instead of admitting the continual regeneration of free sulphuric acid, the author prefers to think that the alcohol, added gradually, acts principally on the two sulphuric ethers, and above all on their products of decomposition,—the sulphonic derivatives, acid and neutral,—which constitute, in great part, the residues, and allow of the explanation of the phenomena observed.

Series 6, vol. vi., No. 2.

Research on the Rapid Estimation of Boric Acid (Preservative) in Milk.—G. Denigès. — The author expresses surprise at Mr. Farrington's recent results on the acidity of boric acid, and he thinks, after having repeated the experiments, that the latter has not obtained the correct results, though the *sense* of his experiments may be correct. By the modified method the author proposes, he claims that his results are exact when the quantity of boric acid present does not exceed 3 grms. per litre, and when the quantity of lactose present in the milk is from 40 to 50 grms. per litre. When these figures are exceeded it is necessary to dilute the milk.

Estimation of Glycerin by Bichromate of Potassium and Sulphuric Acid.—F. Bordas and Sig. de Raczkowski. — Already inserted.

Destruction of Organic Matter in Toxicology.—A. Villiers. — A very convenient mineral ferment, for the destruction of organic matter in toxicological research, can be produced by the aid of salt of manganese. The material to be treated is placed in a flask with dilute hydrochloric acid (3 to 1). Add a few drops of a solution of a manganese salt, and a little nitric acid, which must be renewed as it becomes used up. The mixture must be gently heated. The gases produced are carbonic acid and nearly pure nitrogen, and no disagreeable odours are evolved. Materials such as liver, lungs, &c., are dissolved in a few minutes; muscular fibre takes about an hour, and a fatty mass remains which resists the oxidising action of the mixture, and which appears to contain products of substitution.

On Pseudo-intestinal Calculus.—Marcel Delépine. — The author found, after considerable trouble and groping in the dark, that certain balls which had been sent to him to examine as intestinal calculi were merely undigested balls of bread crumb, which the patient had been in the habit of making and swallowing.

Revue Universelle des Mines et de la Metallurgie.

Series 3, vol. xxxviii., No. 3.

This number contains no matter of chemical interest.

Series 3, vol. xxxix., No. 1.

Saline Deposits of the Plains of Northern Germany.—Franz and Büttgenbach. — There are three rivers in Germany named *Saale*. This name is evidently derived from the numerous saline sources which are to be found in their valleys. In 1837 attempts were made to augment the supply of mineral waters by sinking shafts, but the work was abandoned, as it was found that the waters thus obtained "artificially" were not equal dietetically to those which rose naturally. But in 1861 the commercial value of the potassic salts (*kalisalze*) was recognised, and the industry at Stassfurt soon assumed colossal dimensions. The deposits are of a very complicated nature, and the deposition and formation of the beds must have proceeded and been interrupted many times and at different temperatures. There are at the present time twenty-five different species of deposits known; these are all given in the order of deposition. The most important of these salts are *sythine*, KCl, and *kainite*, $K_2SO_4 \cdot MgSO_4 \cdot MgCl_2 + 6H_2O$. These two salts are found in the ashes of most plants, and are for this reason much sought after as manures. The mean thickness of the beds of the potassic salts is at least 20 metres, and, considering the area covered, the author estimates the quantity available at 10 milliard (10,000,000,000) tons, and, at the present output of 3,000,000 tons a year, the beds will last for 33 centuries.

MISCELLANEOUS.

Obituary.—Professor Victor Meyer died a few days ago. His death seems to have been sudden; but accordant and trustworthy accounts of his end seem as yet wanting.

Sixty-ninth Meeting of the German Association of Natural Science and Medicine.—This Association will meet at Brunswick, after an absence of fifty-six years, on the 20th to the 25th of September, 1897. There will be two principal Sections, the first dealing with Natural Science; it is subdivided into three Sub-sections.

1. Section for Mathematics, Physics, Chemistry, Agriculture, Instruments, &c.
2. Section for Mineralogy, Botany, Zoology, Geography, &c.
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1. Section for General Medicine: Pathology, Anatomy, Surgery, &c.
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3. Section for Anatomical Physiology: Physiological Chemistry, Pharmacology.
4. Section for General Hygiene, Bacteriology, Climatology, Military Sanitation, Veterinary Science, &c.
5. Section for Pharmacy.

A large number of papers have been promised; these, after reading, will be followed by discussions. Though the sections will be all over on the 25th, the meeting will not break up till the 27th, the two extra days being devoted to excursions and social pleasure.

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F. T. STEAVENSON,

Houndgate, Darlington, August 6, 1897. Town Clerk.

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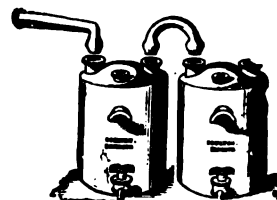
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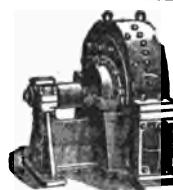
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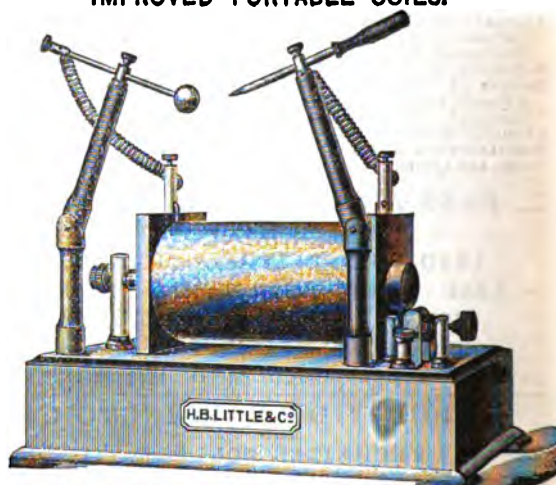
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VOL. LXXVI., No. 1969.

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ONCE more has the Dominion of Canada invited the British Association for the Advancement of Science to hold one of the annual meetings of its members within the Canadian territory; and for a second time has the Association had the honour and pleasure of accepting the proffered hospitality.

In doing so the Association has felt that, if by any possibility the scientific welfare of a locality is promoted by its being the scene of such a meeting, the claims should be fully recognised of those who, though not dwelling in the British Isles, are still inhabitants of that Greater Britain whose prosperity is so intimately connected with the fortunes of the Mother Country.

Here, especially, as loyal subjects of one beloved Sovereign, the sixtieth year of whose beneficent reign has just been celebrated with equal rejoicing in all parts of her Empire; as speaking the same tongue, and as in most instances connected by the ties of one common parentage, we are bound together in all that can promote our common interests.

There is, in all probability, nothing that will tend more to advance those interests than the diffusion of science in all parts of the British Empire, and it is towards this end that the aspirations of the British Association are ever directed, even if in many instances the aim may not be attained.

We are, as already mentioned, indebted to Canada for previous hospitality; but we must also remember that, since the time when we last assembled on this side of the Atlantic, the Dominion has provided the Association with a President, Sir William Dawson, whose name is alike well known in Britain and America, and whose reputation is indeed world-wide. We rejoice that we have still among us the pioneer of American geology, who among other discoveries first made us acquainted with the "Air-breathers of the Coal," the terrestrial, or more properly arboreal, Saurians of the New Brunswick and Nova Scotia Coal-measures.

On our last visit to Canada, in 1884, our place of assembly was Montreal, a city which is justly proud of her McGill University; to-day we meet within the buildings of another of the Universities of this vast Dominion,—and in a city, the absolute fitness of which for such a purpose must have been foreseen by the native Indian tribes when they gave to a small aggregation of huts upon this spot the name of Toronto—"the place of meetings."

Our gathering this year presents a feature of entire novelty and extreme interest, inasmuch as the sister Association of the United States of America,—still mourning the loss of their illustrious President, Professor Cope,—and some other learned societies, have made special arrangements to allow of their members coming here to join us. I need hardly say how welcome their presence is, nor how gladly we look forward to their taking part in

our discussions, and aiding us by interchange of thought. To such a meeting the term "international" seems almost misapplied. It may rather be described as a family gathering, in which our relatives more or less distant in blood, but still intimately connected with us by language, literature, and habits of thought, have spontaneously arranged to take part.

The domain of Science is no doubt one in which the various nations of the civilised world meet upon equal terms, and for which no other passport is required than some evidence of having striven towards the advancement of natural knowledge. Here, on the frontier between the two great English-speaking nations of the world, who is there that does not inwardly feel that anything which conduces to an intimacy between the representatives of two countries, both of them actively engaged in the pursuit of science, may also, through such an intimacy, react on the affairs of daily life, and aid in preserving those cordial relations that have new for so many years existed between the great American Republic and the British Islands, with which her early foundations are indissolubly connected? The present year has witnessed an interchange of courtesies which has excited the warmest feelings of approbation on both sides of the Atlantic. I mean the return to its proper custodians of one of the most interesting of the relics of the Pilgrim Fathers, the Log of the "Mayflower." May this return, trifling in itself, be of happy augury as testifying to the feelings of mutual regard and esteem which animate the hearts both of the donors and of the recipients!

At our meeting in Montreal the President was an investigator who had already attained to a foremost place in the domains of Physics and Mathematics, Lord Rayleigh. In his address he dealt mainly with topics such as Light, Heat, Sound, and Electricity, on which he is one of our principal authorities. His name and that of his fellow-worker, Professor Ramsay, are now and will in all future ages be associated with the discovery of the new element, Argon. Of the ingenious methods by which that discovery was made, and the existence of Argon established, this is not the place to speak. One can only hope that the element will not always continue to justify its name by its inertness.

The claims of such a leader in physical science as Lord Rayleigh to occupy the Presidential chair are self-evident, but possibly those of his successor on this side of the Atlantic are not so immediately apparent. I cannot for a moment pretend to place myself on the same purely scientific level as my distinguished friend and for many years colleague, Lord Rayleigh, and my claims, such as they are, seem to me to rest on entirely different grounds.

Whatever little I may have indirectly been able to do in assisting to promote the advancement of science, my principal efforts have now for many years been directed towards attempting to forge those links in the history of the world, and especially of humanity, that connect the past with the present, and towards tracing that course of evolution which plays as important a part in the physical and moral development of man as it does in that of the animal and vegetable creation.

It appears to me, therefore, that my election to this important post may, in the main, be regarded as a recognition by this Association of the value of Archæology as a science.

Leaving all personal considerations out of question, I gladly hail this recognition, which is, indeed, in full accordance with the attitude already for many years adopted by the Association towards Anthropology, one of the most important branches of true Archæology.

It is no doubt hard to define the exact limits which are to be assigned to Archæology as a science, and Archæology as a branch of History and Belles Lettres. A distinction is frequently drawn between science on the one hand, and knowledge or learning on the other; but translate the terms into Latin, and the distinction at once disappears,

In illustration of this I need only cite Bacon's great work on the "Advancement of Learning," which was, with his own aid, translated into Latin under the title "*De Augmentis Scientiarum*."

It must, however, be acknowledged that a distinction does exist between Archæology proper and what—for want of a better word—may be termed Antiquarianism. It may be interesting to know the internal arrangements of a Dominican convent in the Middle Ages; to distinguish between the different mouldings characteristic of the principal styles of Gothic architecture; to determine whether an English coin bearing the name of Henry was struck under Henry II., Richard, John, or Henry III., or to decide whether some given edifice was erected in Roman, Saxon, or Norman times. But the power to do this, though involving no small degree of detailed knowledge and some acquaintance with scientific methods, can hardly entitle its possessors to be enrolled among the votaries of science.

A familiarity with all the details of Greek and Roman mythology and culture must be regarded as a literary rather than a scientific qualification; and yet when among the records of classical times we come upon traces of manners and customs which have survived for generations, and which seem to throw some rays of light upon the dim past, when history and writing were unknown, we are, I think, approaching the boundaries of scientific Archæology.

Every reader of Virgil knows that the Greeks were not merely orators, but that with a pair of compasses they could describe the movements of the heavens and fix the rising of the stars; but when by modern Astronomy we can determine the heliacal rising of some well-known star, with which the worship in some given ancient temple is known to have been connected, and can fix its position on the horizon at some particular spot, say, three thousand years ago, and then find that the axis of the temple is directed exactly towards that spot, we have some trustworthy scientific evidence that the temple in question must have been erected at a date approximately 1100 years B.C. If on or close to the same site we find that more than one temple was erected, each having a different orientation, these variations, following as they may fairly be presumed to do the changing position of the rising of the dominant star, will also afford a guide as to the chronological order of the different foundations. The researches of Mr. Penrose seem to show that in certain Greek temples, of which the date of foundation is known from history, the actual orientation corresponds with that theoretically deduced from astronomical data.

Sir J. Norman Lockyer has shown that what holds good for Greek temples applies to many of far earlier date in Egypt, though up to the present time hardly a sufficient number of accurate observations have been made to justify us in foreseeing all the instructive results that may be expected to arise from Astronomy coming to the aid of Archæology.

The intimate connection of Archæology with other sciences is in no case so evident as with respect to Geology, for, when considering subjects such as those I shall presently discuss, it is almost impossible to say where the one science ends and the other begins.

By the application of geological methods many archæological questions relating even to subjects on the borders of the historical period have been satisfactorily solved. A careful examination of the limits of the area over which its smaller coins are found has led to the position of many an ancient Greek city being accurately ascertained; while in England it has only been by treating the coins of the Ancient Britons, belonging to a period before the Roman occupation, as if they were actual fossils, that the territories under the dominion of the various kings and princes who struck them have been approximately determined. In arranging the chronological sequence of these coins, the evolution of their types—a process almost as remarkable, and certainly as

well-defined, as any to be found in Nature—has served as an efficient guide. I may venture to add that the results obtained from the study of the morphology of this series of coins were published ten years before the appearance of Darwin's great work on the "Origin of Species."

When we come to the consideration of the relics of the Early Iron and Bronze Ages, the aid of Chemistry has of necessity to be invoked. By its means we are able to determine whether the iron of a tool or weapon is of meteoritic or volcanic origin, or has been reduced from iron-ore, in which case considerable knowledge of metallurgy would be involved on the part of those who made it. With bronze antiquities the nature and extent of the alloys combined with the copper may throw light not only on their chronological position, but on the sources whence the copper, tin, and other metals of which they consist were originally derived. I am not aware of there being sufficient differences in the analyses of the native copper from different localities in the region in which we are assembled, for Canadian Archæologists to fix the sources from which the metal was obtained, which was used in the manufacture of the ancient tools and weapons of copper that are occasionally discovered in this part of the globe.

Like Chemistry, Mineralogy and Petrology may be called to the assistance of Archæology in determining the nature and source of the rocks of which ancient stone implements are made; and, thanks to researches of the followers of those sciences, the old view that all such implements formed of jade and found in Europe must of necessity have been fashioned from material imported from Asia can no longer be maintained. In one respect the Archæologist differs in opinion from the Mineralogist—namely, as to the propriety of chipping off fragments from perfect and highly finished specimens for the purpose of submitting them to microscopic examination.

I have hitherto been speaking of the aid that other sciences can afford to Archæology when dealing with questions that come almost, if not quite, within the fringe of history, and belong to times when the surface of our earth presented much the same configuration as regards the distribution of land and water, and hill and valley, as it does at present, and when, in all probability, the climate was much the same as it now is. When, however, we come to discuss that remote age in which we find the earliest that are at present known of Man's appearance upon earth, the aid of Geology and Palæontology becomes absolutely imperative.

The changes in the surface configuration and in the extent of the land, especially in a country like Britain, as well as the modifications of the fauna and flora since those days, have been such that the Archæologist pure and simple is incompetent to deal with them, and he must either himself undertake the study of these other sciences or call experts in them to his assistance. The evidence that Man had already appeared upon the earth is afforded by stone implements wrought by his hands, and it falls strictly within the province of the Archæologist to judge whether given specimens were so wrought or not; it rests with the Geologist to determine their stratigraphical or chronological position, while the Palæontologist can pronounce upon the age and character of the associated fauna and flora.

If left to himself the Archæologist seems too prone to build up theories founded upon form alone, irrespective of geological conditions. The Geologist, unaccustomed to archæological details, may readily fail to see the difference between the results of the operations of Nature and those of Art, and may be liable to trace the effects of man's handiwork in the chipping, bruising, and wearing which in all ages result from natural forces; but the united labours of the two, checked by those of the Palæontologist, cannot do otherwise than lead towards sound conclusions.

It will perhaps be expected of me that I should on the present occasion bring under review the state of our

present knowledge with regard to the Antiquity of Man; and probably no fitter place could be found for the discussion of such a topic than the adopted home of my venerated friend, the late Sir Daniel Wilson, who first introduced the word "pre-historic" into the English language.

Some among us may be able to call to mind the excitement, not only among men of science but among the general public, when, in 1859, the discoveries of M. Boucher de Perthes and Dr. Rigollot in the gravels of the valley of the Somme, at Abbeville and Amiens, were confirmed by the investigations of the late Sir Joseph Prestwich, myself, and others, and the co-existence of Man with the extinct animals of the Quaternary fauna, such as the mammoth and woolly-haired rhinoceros, was first virtually established. It was at the same time pointed out that these relics belonged to a far earlier date than the ordinary stone weapons found upon the surface, which usually showed signs of grinding or polishing, and that in fact there were two Stone Ages in Britain. To these the terms Neolithic and Palæolithic were subsequently applied by Sir John Lubbock.

The excitement was not less when, at the meeting of this Association at Aberdeen in the autumn of that year, Sir Charles Lyell, in the presence of the Prince Consort, called attention to the discoveries in the valley of the Somme, the site of which he had himself visited, and to the vast lapse of time indicated by the position of the implements in drift-deposits a hundred feet above the existing river.

The conclusions forced upon those who examined the facts on the spot did not receive immediate acceptance by all who were interested in Geology and Archaeology, and fierce were the controversies on the subject that were carried on both in the newspapers and before various learned societies.

It is at the same time instructive and amusing to look back on the discussions of those days. While one class of objectors accounted for the configuration of the flint implements from the gravels by some unknown chemical agency, by the violent and continued gyratory action of water, by fracture resulting from pressure, by rapid cooling when hot or by rapid heating when cold, or even regarded them as aberrant forms of fossil fishes, there were others who, when compelled to acknowledge that the implements were the work of men's hands, attempted to impugn and set aside the evidence as to the circumstances under which they had been discovered. In doing this they adopted the view that the worked flints had either been introduced into the containing beds at a comparatively recent date, or if they actually formed constituent parts of the gravel then that this was a mere modern alluvium resulting from floods at no very remote period.

In the course of a few years the main stream of scientific thought left this controversy behind, though a tendency to cut down the lapse of time necessary for all the changes that have taken place in the configuration of the surface of the earth and in the character of its occupants since the time of the Palæolithic gravels, still survives in the inmost recesses of the hearts of not a few observers.

In his Address to this Association at the Bath meeting of 1864, Sir Charles Lyell struck so true a note that I am tempted to re-produce the paragraph to which I refer:—

"When speculations on the long series of events which occurred in the glacial and post-glacial periods are indulged in, the imagination is apt to take alarm at the immensity of the time required to interpret the monuments of these ages, all referable to the era of existing species. In order to abridge the number of centuries which would otherwise be indispensable, a disposition is shown by many to magnify the rate of change in pre-historic times by investing the causes which have modified the animate and inanimate world with extraordinary and excessive energy. It is related of a great Irish orator of our day that when he was about to contribute somewhat parsimoniously towards a public charity,

he was persuaded by a friend to make a more liberal donation. In doing so he apologised for his first apparent want of generosity by saying that his early life had been a constant struggle with scanty means, and that 'they who are born to affluence cannot easily imagine how long a time it takes to get the chill of poverty out of one's bones.' In like manner we of the living generation, when called upon to make grants of thousands of centuries in order to explain the events of what is called the modern period, shrink naturally at first from making what seems so lavish an expenditure of past time. Throughout our early education we have been accustomed to such strict economy in all that relates to the chronology of the earth and its inhabitants in remote ages, so fettered have we been by old traditional beliefs, that even when our reason is convinced, and we are persuaded that we ought to make more liberal grants of time to the Geologist, we feel how hard it is to get the chill of poverty out of our bones."

Many, however, have at the present day got over this feeling, and of late years the general tendency of those engaged upon the question of the antiquity of the human race has been in the direction of seeking for evidence by which the existence of Man upon the earth could be carried back to a date earlier than that of the Quaternary gravels.

There is little doubt that such evidence will be eventually forthcoming, but, judging from all probability, it is not in Northern Europe that the cradle of the human race will eventually be discovered, but in some part of the world more favoured by a tropical climate, where abundant means of subsistence could be procured, and where the necessity for warm clothing did not exist.

Before entering into speculations on this subject, or attempting to lay down the limits within which we may safely accept recent discoveries as firmly established, it will be well to glance at some of the cases in which implements are stated to have been found under circumstances which raise a presumption of the existence of Man in pre-Glacial, Pliocene, or even Miocene times.

Flint implements of ordinary Palæolithic type have, for instance, been recorded as found in the Eastern Counties of England, in beds beneath the Chalky Boulder Clay; but on careful examination the geological evidence has not to my mind proved satisfactory, nor has it, I believe, been generally accepted. Moreover, the archaeological difficulty that Man, at two such remote epochs as the pre-Glacial and the post-Glacial, even if the term Glacial be limited to the Chalky Boulder Clay, should have manufactured implements so identical in character that they cannot be distinguished apart, seems to have been entirely ignored.

Within the last few months we have had the report of worked flints having been discovered in the late Pliocene Forest Bed of Norfolk; but in that instance the signs of human workmanship upon the flints are by no means apparent to all observers.

But such an antiquity as the Forest Bed is as nothing when compared with that which would be implied by the discoveries of the work of men's hands in the Pliocene and Miocene Beds of England, France, Italy, and Portugal, which have been accepted by some geologists. There is one feature in these cases which has hardly received due attention, and that is the isolated character of the reputed discoveries. Had man, for instance, been present in Britain during the Crag Period, it would be strange indeed if the sole traces of his existence that he left were a perforated tooth of a large shark, the sawn rib of a manatee, and a beaming full face, carved on the shell of a *pedunculus*!

In an address to the Anthropological Section at the Leeds meeting of this Association, in 1890, I dealt somewhat fully with these supposed discoveries of the remains of human art in beds of Tertiary date, and I need not here go further into the question. Suffice it to say that I see no reason why the verdict of "not proven" at which I then arrived should be reversed.

In the case of a more recent discovery in Upper Burma in beds at first pronounced to be Upper Miocene, but subsequently "definitely ascertained to be Pliocene," some of the flints are of purely natural and not artificial origin, so that two questions arise:—First, Were the fossil remains associated with the worked flints or with those of natural forms? And second, Were they actually found in the bed to which they have been assigned, or did they merely lie together on the surface?

Even the *Pithecanthropus erectus* of Dr. Eugene Dubois from Java meets with some incredulous objectors from both the physiological and the geological sides. From the point of view of the latter the difficulty lies in determining the exact age of what are apparently alluvial beds in the bottom of a river valley.

When we return to Palæolithic man, it is satisfactory to feel that we are treading on comparatively secure ground, and that the discoveries of the last forty years in Britain alone enable us to a great extent to re-constitute his history. We may not know the exact geological period when first he settled in the British area, but we have good evidence that he occupied it at a time when the configuration of the surface was entirely different from what it is at present: when the river valleys had not been cut down to anything like their existing depth, when the fauna of the country was of a totally different character from that of the present day, when the extension of the southern part of the island seaward was in places such that the land was continuous with that of the continent, and when in all probability a far more rainy climate prevailed. We have proofs of the occupation of the country by man during the long lapse of time that was necessary for the excavation of the river valleys. We have found the old floors on which his habitations were fixed, we have been able to trace him at work on the manufacture of flint instruments, and by building up the one upon the other the flakes struck off by the primeval workman in those remote times we have been able to reconstruct the blocks of flint which served as his material.

That the duration of the Palæolithic Period must have extended over an almost incredible length of time is sufficiently proved by the fact that valleys, some miles in width and of a depth of from 100 to 150 feet, have been eroded since the deposit of the earliest implement-bearing beds. Nor is the apparent duration of this period diminished by the consideration that the floods which hollowed out the valleys were not in all probability of such frequent occurrence as to teach Palæolithic man by experience the danger of settling too near to the streams, for had he kept to the higher slopes of the valley there would have been but little chance of his implements having so constantly formed constituent parts of the gravels deposited by the floods.

The examination of British cave-deposits affords corroborative evidence of this extended duration of the Palæolithic Period. In Kent's Cavern at Torquay, for instance, we find in the lowest deposit, the breccia below the red cave earth, implements of flint and chert corresponding in all respects with those of the high level and most ancient river gravels. In the cave-earth these are scarcer, though implements occur which also have their analogues in the river deposits; but, what is more remarkable, harpoons of reindeer's horn and needles of bone are present, identical in form and character with those of the caverns of the Reindeer Period in the South of France, and suggestive of some bond of union or identity of descent between the early troglodytes, whose habitations were geographically so widely separated the one from the other.

In a cavern at Creswell Crags, on the confines of Derbyshire and Nottinghamshire, a bone has moreover been found engraved with a representation of parts of a horse in precisely the same style as the engraved bones of the French caves.

It is uncertain whether any of the River-drift specimens belong to so late a date as these artistic cavern-remains;

but the greatly superior antiquity of even these to any Neolithic relics is testified by the thick layer of stalagmite, which had been deposited in Kent's Cavern before its occupation by men of the Neolithic and Bronze Periods.

Towards the close of the period covered by the human occupation of the French caves, there seems to have been a dwindling in the number of the larger animals constituting the Quaternary fauna, whereas their remains are present in abundance in the lower and therefore more recent of the valley gravels. This circumstance may afford an argument in favour of regarding the period represented by the later French caves as a continuation of that during which the old river gravels were deposited, and yet the great change in the fauna that has taken place since the latest of the cave-deposits included in the Palæolithic Period is indicative of an immense lapse of time.

How much greater must have been the time required for the more conspicuous change between the old Quaternary fauna of the river gravels and that characteristic of the Neolithic Period!

As has been pointed out by Prof. Boyd Dawkins, only thirty-one out of the forty-eight well-ascertained species living in the post-Glacial or River-drift Period survived into pre-historic or Neolithic times. We have not, indeed, any means at command for estimating the number of centuries which such an important change indicates; but when we remember that the date of the commencement of the Neolithic or Surface Stone Period is still shrouded in the mist of a dim antiquity, and that prior to that commencement the River-drift period had long come to an end; and when we further take into account the almost inconceivable ages that even under the most favourable conditions the excavation of wide and deep valleys by river action implies, the remoteness of the date at which the Palæolithic Period had its beginning almost transcends our powers of imagination.

We find distinct traces of river action from 100 to 200 feet above the level of existing streams and rivers, and sometimes at a great distance from them; we observe old fresh-water deposits on the slopes of valleys several miles in width; we find that long and lofty escarpments of rock have receded unknown distances since their summits were first occupied by Palæolithic man; we see that the whole side of a wide river valley has been carried away by invasion of the sea, which attacked and removed a barrier of chalk cliffs from 400 to 600 feet in height; we find that what was formerly an inland river has been widened out into an arm of the sea, now the highway of our fleets, and that gravels which were originally deposited in the bed of some ancient river now cap isolated and lofty hills.

And yet, remote as the date of the first known occupation of Britain by man may be, it belongs to what, geologically speaking, must be regarded as a quite recent period, for we are now in a position to fix with some degree of accuracy its place on the geological scale. Thanks to investigations ably carried out at Hoxne in Suffolk, and at Hitchin in Hertfordshire, by Mr. Clement Reid, under the auspices of this Association and of the Royal Society, we know that the implement-bearing beds at those places undoubtedly belong to a time subsequent to the deposit of the Great Chalky Boulder Clay of the Eastern Counties of England. It is, of course, self-evident that this vast deposit, in whatever manner it may have been formed, could not, for centuries after its deposition was complete, have presented a surface inhabitable by man. Moreover, at a distance but little farther north, beds exist which also—though at a somewhat later date—were apparently formed under Glacial conditions. At Hoxne the interval between the deposit of the Boulder Clay and of the implement-bearing beds is distinctly proved to have witnessed at least two noteworthy changes in climate. The beds immediately reposing on the Clay are characterised by the presence of alder in abundance, of hazel, and yew, as well as by that of numerous flower-

ing plants indicative of a temperate climate very different from that under which the Boulder Clay itself was formed. Above these beds characterised by temperate plants, comes a thick and more recent series of strata, in which leaves of the dwarf Arctic willow and birch abound, and which were in all probability deposited under conditions like those of the cold regions of Siberia and North America.

At a higher level and of more recent date than these—from which they are entirely distinct—are the beds containing Palæolithic implements, formed in all probability under conditions not essentially different from those of the present day. However this may be, we have now conclusive evidence that the Palæolithic implements are, in the Eastern Counties of England, of a date long posterior to that of the Great Chalky Boulder Clay.

It may be said, and said truly, that the implements at Hoxne cannot be shown to belong to the beginning rather than to some later stage of the Palæolithic Period.

The changes, however, that have taken place at Hoxne in the surface configuration of the country prove that the beds containing the implements cannot belong to the close of that period.

It must, moreover, be remembered that in what are probably the earliest of the Palæolithic deposits of the Eastern Counties, those at the highest level, near Brandon in Norfolk, where the gravels contain the largest proportion of pebbles derived from Glacial beds, some of the implements themselves have been manufactured from materials not native to the spot, but brought from a distance, and derived in all probability either from the Boulder Clay or from some of the beds associated with it.

We must, however, take a wider view of the whole question, for it must not for a moment be supposed that there are the slightest grounds for believing that the civilisation, such as it was, of the Palæolithic Period originated in the British Isles. We find in other countries implements so identical in form and character with British specimens that they might have been manufactured by the same hands. These occur over large areas in France under similar conditions to those that prevail in England. The same forms have been discovered in the ancient river gravels of Italy, Spain, and Portugal. Some few have been recorded from the north of Africa, and analogous types occur in considerable numbers in the south of that continent. On the banks of the Nile, many hundreds of feet above its present level, implements of the European types have been discovered; while in Somaliland, in an ancient river valley at a great elevation above the sea, Mr. Seton-Karr has collected a large number of implements formed of flint and quartzite, which, judging from their form and character, might have been dug out of the drift deposits of the Somme or the Seine, the Thames or the ancient Solent.

In the valley of the Euphrates implements of the same kind have also been found, and again farther east in the lateritic deposits of Southern India they have been obtained in considerable numbers. It is not a little remarkable, and it is at the same time highly suggestive, that a form of implement almost peculiar to Madras re-appears among implements from the very ancient gravels of the Manzanares at Madrid. In the case of the African discoveries we have as yet no definite Palæontological evidence by which to fix their antiquity; but in the Narbadá Valley of Western India Palæolithic implements of quartzite seem to be associated with a local fauna of Pleistocene age, comprising, like that of Europe, the elephant, hippopotamus, ox, and other mammals of species now extinct. A correlation of the two faunas with a view of ascertaining their chronological relations is beset with many difficulties, but there seems reason for accepting this Indian Pleistocene fauna as in some degree more ancient than the European.

Is this not a case in which the imagination may be fairly invoked in aid of science? May we not from these data attempt in some degree to build up and reconstruct

the early history of the human family? There, in Eastern Asia, in a tropical climate, with the means of subsistence readily at hand, may we not picture to ourselves our earliest ancestors gradually developing from a lowly origin, acquiring a taste for hunting, if not indeed being driven to protect themselves from the beasts around them, and evolving the more complicated forms of tools or weapons from the simpler flakes which had previously served them as knives? May we not imagine that, when once the stage of civilisation denoted by these Palæolithic implements had been reached, the game for the hunter became scarcer, and that his life in consequence assumed a more nomad character? Then, and possibly not till then, may a series of migrations to "fresh woods and pastures new" not unnaturally have ensued, and these, following the usual course of "westward towards the setting sun," might eventually lead to a Palæolithic population finding its way to the extreme borders of Western Europe, where we find such numerous traces of its presence.

How long a term of years may be involved in such a migration it is impossible to say, but that such a migration took place the phenomena seem to justify us in believing. It can hardly be supposed that the process that I have shadowed forth was reversed, and that Man, having originated in North-Western Europe, in a cold climate where clothing was necessary and food scarce, subsequently migrated eastward to India and southward to the Cape of Good Hope! As yet, our records of discoveries in India and Eastern Asia are but scanty; but it is there that the traces of the cradle of the human race are, in my opinion, to be sought, and possibly future discoveries may place upon a more solid foundation the visionary structure that I have ventured to erect.

It may be thought that my hypothesis does not do justice to what Sir Thomas Browne has so happily termed "that great antiquity, America." I am, however, not here immediately concerned with the important Neolithic remains of all kinds with which this great continent abounds. I am now confining myself to the question of Palæolithic man and his origin, and in considering it I am not unmindful of the Trenton implements, though I must content myself by saying that the "turtleback" form is essentially different from the majority of those on the wide dissemination of which I have been speculating, and, moreover, as many here present are aware, the circumstances of the finding of these American implements are still under careful discussion.

Leaving them out of the question for the present, it may be thought worth while to carry our speculations rather further, and to consider the relations in time between the Palæolithic and the Neolithic Periods. We have seen that the stage in human civilisation denoted by the use of the ordinary forms of Palæolithic implements must have extended over a vast period of time if we have to allow for the migration of the primeval hunters from their original home, wherever it may have been in Asia or Africa, to the west of Europe, including Britain. We have seen that, during this migration, the forms of the weapons and tools made from silicious stones had become, as it were, stereotyped, and further, that, during the subsequent extended period implied by the erosion of the valleys, the modifications in the form of the implements and the changes in the fauna associated with the men who used them were but slight.

At the close of the period during which the valleys were being eroded comes that represented by the latest occupation of the caves by Palæolithic man, when both in Britain and in the South of France the reindeer was abundant; but among the stone weapons and implements of that long troglodytic phase of man's history not a single example with the edge sharpened by grinding has as yet been found. All that can safely be said is that the larger implements as well as the larger mammals had become scarcer, that greater power in chipping flint had been attained, that the arts of the engraver and the

sculptor had considerably developed, and that the use of the bow had probably been discovered.

Directly we encounter the relics of the Neolithic Period, often, in the case of the caves lately mentioned, separated from the earlier remains by a thick layer of underlying stalagmite, we find flint hatchets polished at the edge and on the surface, cutting at the broad and not at the narrow end, and other forms of implements associated with a fauna in all essential respects identical with that of the present day.

Were the makers of these polished weapons the direct descendants of Palæolithic ancestors whose occupation of the country was continuous from the days of the old river gravels? or had these long since died out, so that after Western Europe had for ages remained uninhabited, it was re-peopled in Neolithic times by the immigration of some new race of men? Was there, in fact, a "great gulf fixed" between the two occupations? or was there in Europe a gradual transition from the one stage of culture to the other?

It has been said that "what song the Syrens sang, or what name Achilles assumed when he hid himself among women, though puzzling questions, are not beyond all conjecture"; and though the questions now proposed may come under the same category, and must await the discovery of many more essential facts before they receive definite and satisfactory answers, we may, I think, throw some light upon them if we venture to take a few steps upon the seductive if insecure paths of conjecture. So far as I know we have as yet no trustworthy evidence of any transition from the one age to the other, and the gulf between them remains practically unbridged. We can, indeed, hardly name the part of the world in which to seek for the cradle of Neolithic civilisation, though we know that traces of what appear to have been a stone-using people have been discovered in Egypt, and that what must be among the latest of the relics of their industry have been assigned to a date some 3500 to 4000 years before our era. The men of that time had attained to the highest degree of skill in working flint that has ever been reached. Their beautifully made knives and spear-heads seem indicative of a culminating point reached after long ages of experience; but whence these artists in flint came or who they were is at present absolutely unknown, and their handiworks afford no clue to help us in tracing their origin.

Taking a wider survey, we may say that, generally speaking, not only the fauna but the surface configuration of the country were, in Western Europe at all events, much the same at the commencement of the Neolithic Period as they are at the present day. We have, too, no geological indications to aid us in forming any chronological scale.

The occupation of some of the caves in the south of France seems to have been carried on after the erosion of the neighbouring river valleys had ceased, and so far as our knowledge goes these caves offer evidence of being the latest in time of those occupied by Man during the Palæolithic Period. It seems barely possible that, though in the north of Europe there are no distinct signs of such late occupation, yet that, in the south, Man may have lived on, though in diminished numbers; and that in some of the caves, such, for instance, as those in the neighbourhood of Mentone, there may be traces of his existence during the transitional period that connects the Palæolithic and Neolithic Ages. If this were really the case, we might expect to find some traces of a dissemination of Neolithic culture from a North Italian centre, but I much doubt whether any such traces actually exist.

If it had been in that part of the world that the transition took place, how are we to account for the abundance of polished stone hatchets found in Central India? Did Neolithic man return eastward by the same route as that by which in remote ages his Palæolithic predecessor had migrated westward? Would it not be in defiance of all probability to answer such a question in the affirmative?

We have, it must be confessed, nothing of a substantial character to guide us in these speculations; but, pending the advent of evidence to the contrary, we may, I think, provisionally adopt the view that owing to failure of food, climatal changes, or other causes, the occupation of Western Europe by Palæolithic man absolutely ceased, and that it was not until after an interval of long duration that Europe was re-peopled by a race of men immigrating from some other part of the globe where the human race had survived, and in course of ages had developed a higher state of culture than that of Palæolithic man.

I have been carried away by the liberty allowed for conjecture into the regions of pure imagination, and must now return to the realms of fact, and one fact on which I desire for a short time to insist is that of the existence at the present day, in close juxtaposition with our own civilisation, of races of men who, at all events but a few generations ago, lived under much the same conditions as did our own Neolithic predecessors in Europe.

The manners and customs of these primitive tribes and peoples are changing day by day, their languages are becoming obsolete, their myths and traditions are dying out, their ancient processes of manufacture are falling into oblivion, and their numbers are rapidly diminishing, so that it seems inevitable that ere long many of these interesting populations will become absolutely extinct. The admirable Bureau of Ethnology instituted by our neighbours in the United States of America has done much towards preserving a knowledge of the various native races in this vast continent; and here in Canada the annual Archaeological Reports presented to the Minister of Education are rendering good service in the same cause.

Moreover, the Committee of this Association appointed to investigate the physical characters, languages, and industrial and social conditions of the North-Western tribes of the Dominion of Canada is about to present its twelfth and final report, which in conjunction with those already presented will do much towards preserving a knowledge of the habits and languages of those tribes. It is sad to think that Mr. Horatio Hale, whose comprehensive grasp of the bearings of ethnological questions, and whose unremitting labours have so materially conduced to the success of the Committee, should be no longer among us. Although this report is said to be final, it is to be hoped that the Committee may be able to indicate lines upon which future work in the direction of ethnological and archaeological research may be profitably carried on in this part of Her Majesty's dominions.

It is, however, lamentable to notice how little is being or has been officially done towards preserving a full record of the habits, belief, arts, myths, languages, and physical characteristics of the countless other tribes and nations more or less uncivilised which are comprised within the limits of the British Empire. At the meeting of this Association held last year at Liverpool it was resolved by the General Committee "that it is of urgent importance to press upon the Government the necessity of establishing a Bureau of Ethnology for Greater Britain, which by collecting information with regard to the native races within and on the borders of the Empire will prove of immense value to science and to the Government itself." It has been suggested that such a bureau might with the greatest advantage and with the least outlay and permanent expense be connected either with the British Museum or with the Imperial Institute, and the project has already been submitted for the consideration of the Trustees of the former establishment.

The existence of an almost unrivalled ethnological collection in the museum, and the presence there of officers already well versed in ethnological research, seem to afford an argument in favour of the proposed bureau being connected with it. On the other hand, the Imperial Institute was founded with an especial view to its being a centre around which every interest connected with the dependencies of the Empire might gather for information and support. The establishment within the last twelve

months of a Scientific Department within the Institute, with well-appointed laboratories and a highly trained staff, shows how ready are those concerned in its management to undertake any duties that may conduce to the welfare of the outlying parts of the British Empire; a fact of which I believe that Canada is fully aware. The Institute is therefore likely to develop, so far as its scientific department is concerned, into a Bureau of advice in all matters scientific and technical, and certainly a Bureau of Ethnology such as that suggested would not be out of place within its walls.

Wherever such an institution is to be established, the question of its existence must of necessity rest with Her Majesty's Government and Treasury, inasmuch as without funds, however moderate, the undertaking cannot be carried on. I trust that in considering the question it will always be borne in mind that in the relations between civilised and uncivilised nations and races it is of the first importance that the prejudices and especially the religious or semi-religious and caste prejudices of the latter should be thoroughly well known to the former. If but a single "little war" could be avoided in consequence of the knowledge acquired and stored up by the Bureau of Ethnology preventing such a misunderstanding as might culminate in warfare, the cost of such an institution would quickly be saved.

I fear that it will be thought that I have dwelt too long on primeval man and his modern representatives, and that I should have taken this opportunity to discuss some more general subject, such as the advances made in the various departments of science since last this Association met in Canada. Such a subject would no doubt have afforded an infinity of interesting topics on which to dilate. Spectrum analysis, the origin and nature of celestial bodies, photography, the connection between heat, light, and electricity, the practical applications of the latter, terrestrial magnetism, the liquefaction and solidification of gases, the behaviour of elements and compounds under the influence of extreme cold, the nature and uses of the Röntgen rays, the advances in bacteriology and in prophylactic medicine, might all have been passed under review, and to many of my audience would have seemed to possess greater claims to attention than the subject that I have chosen.

It must, however, be borne in mind that most, if not indeed all, of these topics will be discussed by more competent authorities in the various Sections of the Association by means of the Presidential addresses or otherwise. Nor must it be forgotten that I occupy this position as a representative of Archaeology, and am therefore justified in bringing before you a subject in which every member of every race of mankind ought to be interested—the antiquity of the human family and the scenes of its infancy.

Others will direct our thoughts in other directions, but the farther we proceed the more clearly shall we realise the connection and interdependence of all departments of science. Year after year, as meetings of this Association take place, we may also foresee that "many shall run to and fro and knowledge shall be increased." Year after year advances will be made in science, and in reading that Book of Nature that lies ever open before our eyes; successive stones will be brought for building up that Temple of Knowledge of which our fathers and we have laboured to lay the foundations. May we not well exclaim with old Robert Recorde?—

"Oh woorthy temple of Goddes magnificence: Oh throne of glorye and seate of the lorde: thy substance most pure what tounge can describe? thy signes are so wonderous, surmountinge mannes witte, the effects of thy motions so diuers in kinde: so harde for to searche, and worse for to fynde—Thy woorkes are all wonderous, thy cunning unknown: yet seedes of all knowledge in that booke are sowne—And yet in that booke who rightly can reade, to all secrete knowledge it will him straighte leade" (Preface to Robert Recorde's *Castle of Knowledge*, 1556).

ADDRESS TO THE CHEMICAL SECTION OF THE BRITISH ASSOCIATION.

TORONTO, 1897.

By Professor WILLIAM RAMSAY, Ph.D., LL.D., Sc.D., F.R.S.,
President of the Section.

An Undiscovered Gas.

A SECTIONAL address to members of the British Association falls under one of three heads. It may be historical, or actual, or prophetic; it may refer to the past, the present, or the future. In many cases, indeed in all, this classification overlaps. Your former Presidents have given sometimes a historical introduction, followed by an account of the actual state of some branch of our science, and, though rarely, concluding with prophetic remarks. To those who have an affection for the past, the historical side appeals forcibly; to the practical man, and to the investigator engaged in research, the actual, perhaps, presents more charm, while to the general public, to whom novelty is often more of an attraction than truth, the prophetic aspect excites most interest. In this address I must endeavour to tickle all palates; and perhaps I may be excused if I take this opportunity of indulging in the dangerous luxury of prophecy, a luxury which the managers of scientific journals do not often permit their readers to taste.

The subject of my remarks to-day is a new gas. I shall describe to you later its curious properties; but it would be unfair not to put you at once in possession of the knowledge of its most remarkable property—it has not yet been discovered. As it is still unborn, it has not yet been named. The naming of a new element is no easy matter. For there are only twenty-six letters in our alphabet, and there are already over seventy elements. To select a name expressible by a symbol which has not already been claimed by one of the known elements is difficult, and the difficulty is enhanced when it is at the same time required to select a name which shall be descriptive of the properties (or want of properties) of the element.

It is now my task to bring before you the evidence for the existence of this undiscovered element.

It was noticed by Döbereiner, as long ago as 1817, that certain elements could be arranged in groups of three. The choice of the elements selected to form these triads was made on account of their analogous properties, and on the sequence of their atomic weights, which had at that time only recently been discovered. Thus calcium, strontium, and barium formed such a group; their oxides, lime, strontia, and baryta are all easily slaked, combining with water to form soluble lime-water, strontia-water, and baryta-water. Their sulphates are all sparingly soluble, and resemblance had been noticed between their respective chlorides and between their nitrates. Regularity was also displayed by their atomic weights. The numbers then accepted were 20, 42.5, and 65; and the atomic weight of strontium, 42.5, is the arithmetical mean of those of the other two elements, for $(65+20)/2=42.5$. The existence of other similar groups of three was pointed out by Döbereiner, and such groups became known as "Döbereiner's triads."

Another method of classifying the elements, also depending on their atomic weights, was suggested by Pettenkofer, and afterwards elaborated by Kremers, Gladstone, and Cooke. It consisted in seeking for some expression which would represent the differences between the atomic weights of certain allied elements. Thus, the difference between the atomic weight of lithium, 7, and sodium, 23, is 16; and between that of sodium and of potassium, 39, is also 16. The regularity is not always so conspicuous; Dumas, in 1857, contrived a somewhat complicated expression which, to some extent, exhibited regularity in the atomic weights of fluorine, chlorine, bro-

mine, and iodine; and also of nitrogen, phosphorus, arsenic, antimony, and bismuth.

The upshot of these efforts to discover regularity was that, in 1864, Mr. John Newlands, having arranged the elements in eight groups, found that when placed in the order of their atomic weights, "the eighth element, starting from a given one, is a kind of repetition of the first, like the eighth note of an octave in music." To this regularity he gave the name "The Law of Octaves."

The development of this idea, as all chemists know, was due to the late Professor Lothar Meyer, of Tübingen, and to Professor Mendeléeff, of St. Petersburg. It is generally known as the "Periodic Law." One of the simplest methods of showing this arrangement is by means of a cylinder divided into eight segments by lines drawn parallel to its axis; a spiral line is then traced round the cylinder, which will, of course, be cut by these lines eight times at each revolution. Holding the cylinder vertically, the name and atomic weight of an element is written at each intersection of the spiral with a vertical line, following the numerical order of the atomic weights. It will be found, according to Lothar Meyer and Mendeléeff, that the elements grouped down each of the vertical lines form a natural class; they possess similar properties, form similar compounds, and exhibit a graded relationship between their densities, melting-points, and many of their other properties. One of these vertical columns, however, differs from the others, inasmuch as on it there are three groups, each consisting of three elements with approximately equal atomic weights. The elements in question are iron, cobalt, and nickel; palladium, rhodium, and ruthenium; and platinum, iridium, and osmium. There is apparently room for a fourth group of three elements in this column, and it may be a fifth. And the discovery of such a group is not unlikely, for when this table was first drawn up Professor Mendeléeff drew attention to certain gaps, which have since been filled up by the discovery of gallium, germanium, and others.

The discovery of argon at once raised the curiosity of Lord Rayleigh and myself as to its position in this table. With a density of nearly 20, if a diatomic gas, like oxygen and nitrogen, it would follow fluorine in the periodic table; and our first idea was that argon was probably a mixture of three gases, all of which possessed nearly the same atomic weights, like iron, cobalt, and nickel. Indeed, their names were suggested, on this supposition, with patriotic bias, as Anglium, Scotium, and Hibernium! But when the ratio of its specific heats had, at least in our opinion, unmistakably shown that it was molecularly monatomic, and not diatomic, as at first conjectured, it was necessary to believe that its atomic weight was 40, and not 20, and that it followed chlorine in the atomic table, and not fluorine. But here arises a difficulty. The atomic weight of chlorine is 35.5, and that of potassium, the next element in order in the table, is 39.1; and that of argon, 40, follows, and does not precede, that of potassium, as it might be expected to do. It still remains possible that argon, instead of consisting wholly of monatomic molecules, may contain a small percentage of diatomic molecules; but the evidence in favour of this supposition is, in my opinion, far from strong. Another possibility is that argon, as at first conjectured, may consist of a mixture of more than one element; but unless the atomic weight of one of the elements in the supposed mixture is very high, say 82, the case is not bettered, for one of the elements in the supposed trio would still have a higher atomic weight than potassium. And very careful experiments, carried out by Dr. Norman Collie and myself, on the fractional diffusion of argon, have disproved the existence of any such element with high atomic weight in argon, and, indeed, have practically demonstrated that argon is a simple substance, and not a mixture.

The discovery of helium has thrown a new light on this subject. Helium, it will be remembered, is evolved on heating certain minerals, notably those containing

uranium; although it appears to be contained in others in which uranium is not present, except in traces. Among these minerals are clèdeite, monazite, fergusonite, and a host of similar complex mixtures, all containing rare elements, such as niobium, tantalum, yttrium, cerium, &c. The spectrum of helium is characterised by a remarkably brilliant yellow line, which had been observed as long ago as 1868 by Professors Frankland and Lockyer in the spectrum of the sun's chromosphere, and named "helium" at that early date.

The density of helium proved to be very close to 2.0, and, like argon, the ratio of its specific heat showed that it, too, was a monatomic gas. Its atomic weight therefore is identical with its molecular weight, viz., 4.0, and its place in the periodic table is between hydrogen and lithium, the atomic weight of which is 7.0.

The difference between the atomic weights of helium and argon is thus 36, or 40-4. Now there are several cases of such a difference. For instance, in the group the first member of which is fluorine we have—

Fluorine..	..	..	..	..	19	16.5
Chlorine..	..	..	..	..	35.5	19.5
Manganese ..	..	..	..	..	55	

In the oxygen group—

Oxygen ..	..	..	..	..	16	16
Sulphur ..	..	..	..	..	32	20.3
Chromium ..	..	..	..	..	52.3	

In the nitrogen group—

Nitrogen ..	..	..	..	..	14	17
Phosphorus ..	..	..	..	..	31	20.4
Vanadium ..	..	..	..	..	51.4	

And in the carbon group—

Carbon ..	..	..	..	..	12	16.3
Silicon ..	..	..	..	..	28.3	19.8
Titanium ..	..	..	..	..	48.1	

These instances suffice to show that approximately the differences are 16 and 20 between consecutive members of the corresponding groups of elements. The total differences between the extreme members of the short series mentioned are—

Manganese—Fluorine ..	..	..	..	36
Chromium—Oxygen ..	..	..	..	36.3
Vanadium—Nitrogen ..	..	..	..	37.4
Titanium—Carbon ..	..	..	..	36.1

This is approximately the difference between the atomic weights of helium and argon, 36.

There should, therefore, be an undiscovered element between helium and argon, with an atomic weight 16 units higher than that of helium, and 20 units lower than that of argon, namely 20. And if this unknown element, like helium and argon, should prove to consist of monatomic molecules, then its density should be half its atomic weight, 10. And pushing the analogy still farther, it is to be expected that this element should be as indifferent to union with other elements as the two allied elements.

My assistant, Mr. Morris Travers, has indefatigably aided me in a search for this unknown gas. There is a proverb about looking for a needle in a haystack; modern science, with the aid of suitable magnetic appliances, would, if the reward were sufficient, make short work of that proverbial needle. But here is a supposed unknown gas, endowed no doubt with negative properties, and the whole world to find it in. Still, the attempt had to be made.

We first directed our attention to the sources of helium—minerals. Almost every mineral which we could obtain was heated in a vacuum, and the gas which was evolved examined. The results are interesting. Most minerals give off gas when heated, and the gas contains, as a rule, a considerable amount of hydrogen, mixed with

carbonic acid, questionable traces of nitrogen, and carbonic oxide. Many of the minerals, in addition, gave helium, which proved to be widely distributed, though only in minute proportion. One mineral—malacone—gave appreciable quantities of argon; and it is noteworthy that argon was not found except in it (and, curiously, in much larger amount than helium), and in a specimen of meteoric iron. Other specimens of meteoric iron were examined, but were found to contain mainly hydrogen, with no trace of either argon or helium. It is probable that the sources of meteorites might be traced in this manner, and that each could be relegated to its particular swarm.

Among the minerals examined was one to which our attention had been directed by Professor Lockyer, named eliasite, from which he said that he had extracted a gas in which he had observed spectrum lines foreign to helium. He was kind enough to furnish us with a specimen of this mineral, which is exceedingly rare, but the sample which we tested contained nothing but undoubted helium.

During a trip to Iceland in 1895, I collected some gas from the boiling springs there; it consisted, for the most part, of air, but contained somewhat more argon than is usually dissolved when air is shaken with water. In the spring of 1896 Mr. Travers and I made a trip to the Pyrenees to collect gas from the mineral springs of Caunterets, to which our attention had been directed by Dr. Bouchard, who pointed out that these gases are rich in helium. We examined a number of samples from the various springs, and confirmed Dr. Bouchard's results, but there was no sign of any unknown lines in the spectrum of these gases. Our quest was in vain.

We must now turn to another aspect of the subject. Shortly after the discovery of helium, its spectrum was very carefully examined by Professors Runge and Paschen, the renowned spectroscopists. The spectrum was photographed, special attention being paid to the invisible portions, termed the "ultra-violet" and "infra-red." The lines thus registered were found to have a harmonic relation to each other. They admitted of division into two sets, each complete in itself. Now, a similar process had been applied to the spectrum of lithium and to that of sodium, and the spectra of these elements gave only one series each. Hence, Professors Runge and Paschen concluded that the gas, to which the provisional name of helium had been given, was, in reality, a mixture of two gases, closely resembling each other in properties. As we know no other elements with atomic weights between those of hydrogen and lithium, there is no chemical evidence either for or against this supposition. Professor Runge supposed that he had obtained evidence of the separation of these imagined elements from each other by means of diffusion; but Mr. Travers and I pointed out that the same alteration of spectrum, which was apparently produced by diffusion, could also be caused by altering the pressure of the gas in the vacuum tube; and shortly after Professor Runge acknowledged his mistake.

These considerations, however, made it desirable to subject helium to systematic diffusion, in the same way as argon had been tried. The experiments were carried out in the summer of 1896 by Dr. Collie and myself. The result was encouraging. It was found possible to separate helium into two portions of different rates of diffusion, and consequently of different density by this means. The limits of separation, however, were not very great. On the one hand, we obtained gas of a density close on 2.0; and on the other, a sample of density 2.4 or thereabouts. The difficulty was increased by the curious behaviour, which we have often had occasion to confirm, that helium possesses a rate of diffusion too rapid for its density. Thus, the density of the lightest portion of the diffused gas, calculated from its rate of diffusion, was 1.874; but this corresponds to a real density of about 2.0. After our paper, giving an account of these experiments, had been

published, a German investigator, Herr A. Hagenbach, repeated our work and confirmed our results.

The two samples of gas of different density differ also in other properties. Different transparent substances differ in the rate at which they allow light to pass through them. Thus, light travels through water at a much slower rate than through air, and at a slower rate through air than through hydrogen. Now Lord Rayleigh found that helium offers less opposition to the passage of light than any other substance does, and the heavier of the two portions into which helium had been split offered more opposition than the lighter portion. And the retardation of the light, unlike what has usually been observed, was nearly proportional to the densities of the samples. The spectrum of these two samples did not differ in the minutest particular; therefore it did not appear quite out of the question to hazard the speculation that the process of diffusion was instrumental, not necessarily in separating two kinds of gas from each other, but actually in removing light molecules of the same kind from heavy molecules. This idea is not new. It had been advanced by Prof. Schützenberger (whose recent death all chemists have to deplore), and later, by Sir William Crookes, that what we term the atomic weight of an element is a mean; that when we say that the atomic weight of oxygen is 16, we merely state that the average atomic weight is 16; and it is not inconceivable that a certain number of molecules have a weight somewhat higher than 32, while a certain number have a lower weight.

We therefore thought it necessary to test this question by direct experiment with some known gas; and we chose nitrogen, as a good material with which to test the point. A much larger and more convenient apparatus for diffusing gases was built by Mr. Travers and myself, and a set of systematic diffusions of nitrogen was carried out. After thirty rounds, corresponding to 180 diffusions, the density of the nitrogen was unaltered, and that of the portion which should have diffused most slowly, had there been any difference in rate, was identical with that of the most quickly diffusing portion—i.e., with that of the portion which passed first through the porous plug. This attempt, therefore, was unsuccessful; but it was worth carrying out, for it is now certain that it is not possible to separate a gas of undoubted chemical unity into portions of different density by diffusion. And these experiments rendered it exceedingly improbable that the difference in density of the two fractions of helium was due to separation of light molecules of helium from heavy molecules.

(To be continued).

BACTERIOLOGICAL STUDY OF AMBERGRIS.

By H. BEAUREGARD.

I HAVE formerly shown, in concert with the regretted Professor G. Gouchet, that ambergris is an interesting calculus which is developed and has its seat in the rectum of the sperm whale.

This calculus, composed of crystals of ambrine mixed with a larger or smaller amount of black pigment derived from the rectal lining, contains also star-coral debris. When it is fresh, i.e., when it is just extracted from the rectum by the fishermen, it is of a soft consistency, and its odour is not at all agreeable on account of its predominant excrementitious character. But after being preserved for some years in an air-tight tin case it is gradually freed from this excrementitious odour, though losing little of its weight, and retains merely a delicate perfume *sui generis*, which gives it such a value that it reaches the price of from 3000 to 7000 frs. per kilometre. This is not a case of slow desiccation, and cannot be imitated or accelerated by the withdrawal of water. The change is due to a microbe for which the author proposes

the name *Spirillum recti Physteris*. As regards polymorphism this microbe is comparable to the spirillum of cholera. It is probable that the destruction of the faecal odour and the genesis of the delicate perfume are microbial phenomena. It remains to determine if the spirillum in question is pathogenous, at least for terrestrial animals. —*Comptes Rendus*, cxv., p. 254.

NOTICES OF BOOKS.

Twenty-first Annual Report of Her Majesty's Inspectors of Explosives; being their Annual Report for the Year 1896. Pp. 184. London: Eyre and Spottiswoode. 1897.

DURING the past year a further modification of the Explosives Act of 1875 has taken place, the Orders in Council relating to premises registered for mixed explosives having been repealed, and a new Order (No. 16) substituted. The main object of this new Order was to give relief in the storage of small-arm nitro-compounds.

The number of factories making explosives has had a further increase, five new ones having been started since 1895, while seven applications for new licenses are still under consideration. In the case of magazines there is a net increase of two, and there are nine further applications under consideration.

Every factory has been visited by the Inspectors at least once a year, and nearly one-half of them twice; and it is satisfactory to note that there has been no falling off in the high standard previously attained in the great majority of these establishments, while some which had been behind have been much improved.

A striking example of the great improvement in the conduct of the factories, since the passing of the Explosives Act, is to be found in the fact that only one death from accidents by fire or explosion in any factory has occurred during the year, and this in spite of the large increase in the number of factories, which is now 139, though three are temporarily closed.

In Appendix A will be found a table of all factories now in existence, with the classes of explosives authorised to be manufactured therein; and Appendix E gives their distribution about the country. There have been six additions to the list of authorised explosives during the year, particulars of which are set forth in Appendix D (1). As already explained in a previous Report, all nitro-cotton is now admitted to be explosive; collodion cotton, therefore, appears on the authorised list, but the exemptions—when it is in solution in alcohol and ether, or wet with methylated spirit, and enclosed in air-tight cases, still hold good.

Of the 358 samples taken during the year, and examined by Dr. Dupré, only 32 were rejected, and of these 9 were amorces.

One conviction has been obtained during the year for manufacturing fireworks containing sulphur and chlorate, contrary to the provisions of the Order in Council; one for manufacturing amorces and throw-downs with an excess of composition; while at two factories explosives have been placed under seizure. Although, as we mentioned above, there has been only one death from accident, there have been 46 accidents from one cause and another, by which in addition 26 persons were injured: details are to be found in Appendix W, and in the accident section of this Report.

It is satisfactory to see that no case of illegal manufacture has come under the notice of the Inspectors during the year.

There have been 416 visits paid during the year to the 384 magazines in the United Kingdom, and in every case the Inspectors were well satisfied, and there has been no accident by fire or explosion at any one of them during this period. There has been no fault found with the

character of the packing of explosives,—as a rule it has been excellent.

The amount of foreign nitro-glycerin compounds imported during 1896 shows a considerable increase over that imported in 1895; the increase being from 880,070 lbs. to 1,259,200 lbs., most of which was in carbonite: this explosive now heads the list, gelatin-dynamite no longer holding first place.

The Inspectors have no reason to modify their previously expressed opinion that their powers of search and seizure are ample and satisfactory. The total cases of seizure of all sorts was 15, the average for the previous ten years being 24.9.

As usual, the registered premises have been the most troublesome branch of the Inspector's duties; their number, their distribution, and the apparently extreme ignorance of the occupiers in many cases renders it difficult to make much impression on them as a mass,—still it is exceptional now to find large excesses of explosives stored in them.

A detailed list of all the accidents which came under the notice of the Inspectors is given; perhaps the most interesting of these was the one at the Arklow Factory on December 1st. A charge of glycerin was being nitrated in the ordinary way, and it is believed that about 60 lbs. of nitro-glycerin was in the nitrator. The acids had been blown in, and cooled, and the injection of glycerin had proceeded for about ten minutes, when suddenly, without warning, the charge fumed off. The connections to the drowning tank were immediately opened, but it was subsequently found that no nitro-glycerin was among the water and acids in the drowning tank, this leading to the conclusion that the whole of the 60 lbs. of nitro-glycerin had fumed off, in the few seconds from the first establishment of the action to the escape of the charge into the drowning tank; and as the thermometer did not show any increase of temperature—it stood at 12° C.—it is assumed that a leak in one of the coils had occurred and caused a local rise of temperature: such a leak was found after the accident, though it is confidently stated that the coils were tested by air pressure before the commencement of the operation, and that no leak existed then. Fortunately there was no loss of life or personal injury.

Among foreign explosions, one of the most tremendous of modern times occurred on the 19th of February, at Johannesburg, South Africa, when no less than 55 tons of blasting gelatin, contained in ten trucks, exploded on the railway, about 300 yards from Johannesburg station, under circumstances which in our opinion amount to criminal negligence, and almost inconceivable stupidity. The trucks containing the blasting gelatin had been standing for three and a half days, without supervision, in the blazing sun, in a place surrounded by human habitations; dynamite and detonators were carried in the same truck; the trucks were so badly packed that several cases of dynamite had fallen off in transit, and, further, this particular consignment was not packed in cases thick enough and strong enough according to the law. It is not so much a matter of surprise that, when a train that was being shunted ran into the trucks, owing to the points being wrongly turned, an explosion took place, as it is, that in this maze of ignorance and carelessness many other explosions had not already occurred.

A Course of Practical Chemistry. By M. M. PATTISON MUIR, M.A. Part I.—Elementary. Pp. 318. London: Longmans, Green, and Co. 1897.

THE author's experience as a teacher for twenty-five years has convinced him that qualitative analysis cannot be properly learned at an early stage of a course of practical chemistry. He therefore, in this volume, lays more stress on the simple work of preparations, and experiments on the reactions of the chief inorganic compounds, before going on to actual analysis. This is as it should

be; an apprentice to a carpenter learns to saw straight before he begins to make a cabinet.

The volume now before us (Part I.) is divided into three sections, and these again are subdivided into fifty-two lessons.

Section I. deals with experiments on chemical change; preparations of various compounds; and the reactions of acids, alkalies, and salts—in which all the ordinary elementary work of a chemical laboratory is fully described. There are also some notes preceding the actual lessons giving useful hints to a beginner, straight and to the point. E.g., "Note II. Always read the whole of the directions before beginning an experiment." "When you are told to 'heat gently,' this means use a small Bunsen flame for heating, and keep the flame at some distance from the apparatus to be heated."

Section II. concerns volumetric estimations of acids, alkalies, iron salts, chlorides, iodine, &c., the necessary standard solutions being given.

In Section III. we arrive at qualitative analysis, beginning with experiments to illustrate the methods used for dividing the metals into groups, and distinguishing the several metals in these same groups, going all through the six groups, and finally coming to the examination of a solid by dry or blowpipe tests.

The Appendices, of which there are five, contain, amongst other information, instructions for making simple apparatus, the usual tables, list of reagents and their preparation, standard solutions, and a list of substances suitable for the various exercises. The Index is good and complete, and the whole arrangement of the book seems to be very convenient.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxv., No. 3, July 19, 1897.

The Minister of Public Instruction communicated the text of the decree by which the President of the French Republic approves the election of Prof. Virchow as a Foreign Associate *vice* M. Tchebicheff, deceased.

Researches on the State of Elements other than Carbon in Cast-irons and Steels.—Ad. Carnot and M. Goutal.—Will be inserted in full.

Phenomenon of the Electric Arc.—A. Blondel.—This paper requires the accompanying diagram.

Action of Electric Charges on the property of Discharge created by the X Rays in the Air.—E. Villari.—It is known that gases traversed by the X rays acquire the property of discharging electrified conductors. I have demonstrated that they retain this property, though in a less degree after having traversed tubes of glass or of lead of 20 metres in length, or even upwards. I have arrived at the following conclusions:—Air traversed by the X rays and blown against the end of a wire in its natural state retains entirely its discharging power undiminished. If blown against the end of an electrified wire (+) it entirely loses its power of discharging an electroscope (+) having the same sign as that of the wire. Air traversed by the X rays and drawn against the approximating ends of two wires having opposite charges loses all discharging power. The air traversed by X rays, on passing by an ozoniser, acts in these experiments as if its molecules had opposite charges by which it can discharge electrified bodies.

Properties of Gases Traversed by the X Rays, and on the Properties of Luminescence on Photographic Bodies.—G. Sagnac.

The Spectrum of Carbon.—A. de Gramont.—This paper requires the accompanying diagram.

Action of Cupric Hydrate upon Solutions of Silver Nitrate and Basic Argentic Cuprate.—Paul Sabatier.—The formation of a basic mixed argento-cupric salt is not confined to the nitrates. An analogous production ensues on setting out from the sulphates, chlorates, and hyposulphates, as the author intends to show in a future communication.

Hydrobenzamide, Amarine, and Lophine.—Marcel Delépine.—An elaborate thermo-chemical memoir, in which we recognise the constant universality of the thermic laws regulating the chemical transformations of the complicated molecules.

New Syntheses by means of Cyano-succinic Ether.—L. Barth.—Not adapted for useful abstraction.

Certain Compounds of Phenylhydrazine and Metallic Nitrates.—J. Moitessier.—The nitrates of the magnesian metals combine directly with phenylhydrazine like the corresponding haloid salts, yielding crystalline compounds presenting the reactions of phenylhydrazine and those of the metals which they contain. They deflagrate if heated, as do the nitrates in presence of carbon, leaving a more or less abundant residue of metallic oxide.

On the Aloines.—E. Léger.—The aloines may be divided into two groups: the first including barbaloine, socaloine, zanaloine, and curaraloine; whilst the second contains a single representative, mataloine.

MISCELLANEOUS.

Action of X Rays upon the Temperature of Animals.—L. Lecerclé.—Exposure to X rays modifies the cutaneous and rectal temperatures both in the same direction. Under their influence these temperatures sink at first, and then rise above the initial degree.—*Comptes Rendus*, cxv., No. 4.

Actino-Electric Effects of the Röntgen Rays.—S. Puggenheimer.—The author has obtained the following results:—If we plunge two identical electrodes into a liquid and expose the *n*-th one to the X rays there is set up a current which ordinarily flows from the plate exposed to the X rays to the *n*-th of the external circuit. The intensity of the current depends on the intensity of the radiation.—*Comptes Rendus*, cxv., No. 1.

On the Applications of Electrolysis to Organic Chemistry.—L. Gourwitsch.—The action of electrolysis on the salts of the fatty acids nearly always gives rise to the formation of alcohols, ethers, acids, &c., in quantities variable according to the conditions of the experiment. A large number of experiments made by Loeb, Bourgoin, Kolbe, Brown and Walker, Mulliken, and many others, are quoted, and the "mechanism" of the reactions are discussed. Iodoform is now being prepared electrolytically by substitution, by passing the current through a solution of iodide of potassium in alcohol or aqueous acetone, and neutralising the excess of potash formed by carbonic acid; the iodine and the potash formed by the action of the current react with the solvent, and form crystals of perfectly pure iodoform. The nitrified derivatives of the aromatic series serve best for studying the reduction by electrolysis. In 1882 Kendall patented the manufacture of aniline and toluidine by the action of the electric current on mixtures of nitrobenzene and nitrotoluene with concentrated sulphuric acid; but the return was very bad, and the process was of no practical value. Twelve years later the subject was again taken up, and it was shown that aniline was formed even in acid solution. It is interesting to note that the platinum electrodes were strongly attacked in the experiments on carbamate of ammonia, a complicated platino-ammoniacal base being formed.—*Moniteur Scientifique*, xl., Part 1.

Australasian Association for the Advancement of Science.—The objects of this Association, which is now about to commence its Seventh Session, are to give a stronger impulse and a more systematic direction to scientific enquiry, and to promote the intercourse of those who cultivate science in different parts of the Australasian Colonies and in other countries. The Seventh Session will commence in Sydney, on Monday, January 6th next, and will last till the next Thursday, under the presidency of Professor A. Liversidge, M.A., LL.D., F.R.S. The work of the Association will be divided into ten sections, devoted to all the principal scientific subjects. The subscription to become an Interim Member is £1, or £10 to become a Life Member. Excursions will be organised to places of interest, such as the various mining districts, the Zenolan and Wambeyan Caves, the Blue Mountains, &c. All particulars can be obtained from the Hon. Secretaries, Australasian Association for the Advancement of Science, The University, Sydney.

NOTES AND QUERIES.

* Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Cooling Water.—Can any reader give me particulars of a simple, cheap, and effective means of cooling (and the colder the better) a fairly large quantity of water, say three or four pails full, for use in a hot climate? The water is not for cooking or washing purposes.—D. J. P.

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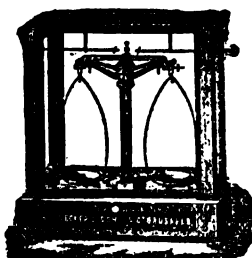
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FIG. 11. No. 91.

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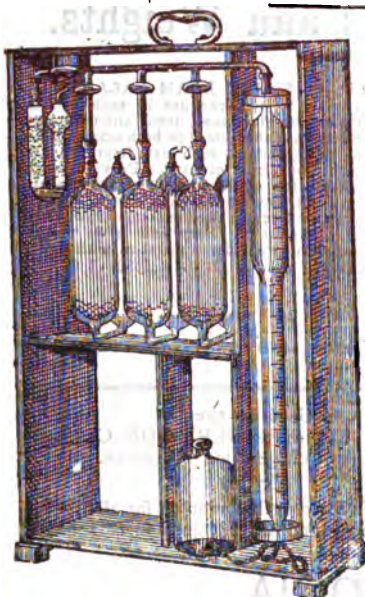
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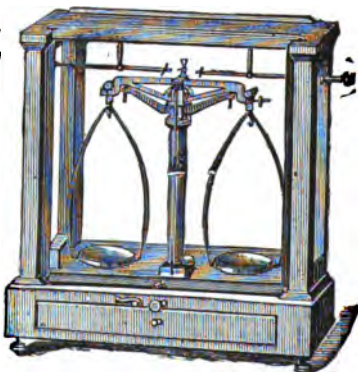


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ADDRESS TO THE CHEMICAL SECTION

By Professor WILLIAM RAMSAY, F.R.S., F.L.S., F.R.S.,
President of the Section.

(Concluded from p. 96.)

The apparatus used for diffusion had a capacity of about two litres. It was filled with helium, and the operation of diffusion was carried through thirty times. There were six reservoirs, each full of gas, and each was separated into two by diffusion. To the heavier portion of one lot, the lighter portion of the next was added, and in this manner all six reservoirs were successively passed through the diffusion apparatus. This process was carried out thirty times, each of the six reservoirs having had its gas diffused each time, thus involving 180 diffusions. After this process, the density of the more quickly diffusing gas was reduced to 2.02, while that of the less quickly diffusing had increased to 2.27. The light portion on re-diffusion hardly altered in density, while the heavier portion, when divided into three portions by diffusion, showed a considerable difference in density between the first third and the last third. A similar set of operations was carried out with a fresh quantity of helium, in order to accumulate enough gas to obtain a sufficient quantity for a second series of diffusions. The more quickly diffusing portions of both gases were mixed and re-diffused. The density of the lightest portion of these gases was 1.98; and after other 15 diffusions, the density of the lightest portion had not decreased. The end had been reached; it was not possible to obtain a lighter portion by diffusion. The density of the main body of this gas is therefore 1.98; and its refractivity, air being taken as unity, is 0.1245. The spectrum of this portion does not differ in any respect from the usual spectrum of helium.

As re-diffusion does not alter the density or the refractivity of this gas, it is right to suppose that either one definite element has now been isolated, or that, if there are more elements than one present, they possess the same, or very nearly the same, density and refractivity. There may be a group of elements, say three, like iron, cobalt, and nickel; but there is no proof that this idea is correct, and the simplicity of the spectrum would be an argument against such a supposition. This substance, forming by far the larger part of the whole amount of the gas, must, in the present state of our knowledge, be regarded as pure helium.

On the other hand, the heavier residue is easily altered in density by re-diffusion, and this would imply that it consists of a small quantity of a heavy gas mixed with a large quantity of the light gas. Repeated re-diffusion convinced us that there was only a very small amount of the heavy gas present in the mixture. The portion which contained the largest amount of heavy gas was found to have the density 2.275, and its refractive index was found to be 0.1333. On re-diffusing this portion of gas until only a trace sufficient to fill a Plücker's tube was left, and then examining the spectrum, no unknown lines could be detected, but, on interposing a jar and spark gap, the well-known blue lines of argon became visible; and even without the jar the red lines of argon and the two green groups were distinctly visible. The amount of

argon present, calculated from the density, was 1.64 per cent, and from the refractivity 1.14 per cent. The conclusion had therefore to be drawn that the heavy constituent of helium, as it comes off the minerals containing it, is nothing new, but, so far as can be made out, merely a small amount of argon.

If, then, there is a new gas in what is generally termed helium, it is mixed with argon, and it must be present in extremely minute traces. As neither helium nor argon has been induced to form compounds, there does not appear to be any method, other than diffusion, for isolating such a gas, if it exists, and that method has failed in our hands to give any evidence of the existence of such a gas. It by no means follows that the gas does not exist; the only conclusion to be drawn is that we have not yet stumbled on the material which contains it. In fact, the haystack is too large and the needle too inconspicuous. Reference to the periodic table will show that between the elements aluminium and indium there occurs gallium, a substance occurring only in the minutest amount on the earth's surface; and following silicon, and preceding tin, appears the element germanium, a body which has as yet been recognised only in one of the rarest of minerals, argyrodite. Now, the amount of helium in fergusonite, one of the minerals which yields it in reasonable quantity, is only 33 parts by weight in 100,000 of the mineral; and it is not improbable that some other mineral may contain the new gas in even more minute proportion. If, however, it is accompanied in its still undiscovered source by argon and helium, it will be a work of extreme difficulty to effect a separation from these gases.

In these remarks it has been assumed that the new gas will resemble argon and helium in being indifferent to the action of reagents, and in not forming compounds. This supposition is worth examining. In considering it, the analogy with other elements is all that we have to guide us.

We have already paid some attention to several triads of elements. We have seen that the differences in atomic weights between the elements fluorine and manganese, oxygen and chromium, nitrogen and vanadium, carbon and titanium, is in each case approximately the same as that between helium and argon, viz., 36. If elements further back in the periodic table be examined, it is to be noticed that the differences grow less, the smaller the atomic weights. Thus, between boron and scandium the difference is 33; between beryllium (glucinum) and calcium, 31; and between lithium and potassium, 32. At the same time, we may remark that the elements grow liker each other, the lower the atomic weights. Now, helium and argon are very like each other in physical properties. It may be fairly concluded, I think, that in so far they justify their position. Moreover, the pair of elements which show the smallest difference between their atomic weights is beryllium and calcium; there is a somewhat greater difference between lithium and potassium. And it is in accordance with this fragment of regularity that helium and argon show a greater difference. Then again, sodium, the middle element of the lithium triad, is very similar in properties both to lithium and potassium; and we might, therefore, expect that the unknown element of the helium series should closely resemble both helium and argon.

Leaving now the consideration of the new element, let us turn our attention to the more general question of the atomic weight of argon, and its anomalous position in the periodic scheme of the elements. The apparent difficulty is this:—The atomic weight of argon is 40; it has no power to form compounds, and thus possesses no valency; it must follow chlorine in the periodic table, and precede potassium; but its atomic weight is greater than that of potassium, whereas it is generally contended that the elements should follow each other in the order of their atomic weights. If this contention is correct, argon should have an atomic weight smaller than 40.

Let us examine this contention. Taking the first row of elements, we have:—

Li=7, Be=9.8, B=11, C=12, N=14, O=16, F=19,
? = 20.

The differences are:—

2.8, 1.2, 1.0, 2.0, 2.0, 3.0, 1.0.

It is obvious that they are irregular. The next row shows similar irregularities. Thus:—

(? = 20, Na = 23, Mg = 24.3, Al = 27, Si = 28, P = 31, S = 32,
Cl = 35.5, A = 40.

And the differences:—

3.0, 1.3, 2.7, 1.0, 3.0, 1.0, 3.5, 4.5.

The same irregularity might be illustrated by a consideration of each succeeding row. Between argon and the next in order, potassium, there is a difference of .09; that is to say, argon has a higher atomic weight than potassium by .09 unit, whereas it might be expected to have a lower one, seeing that potassium follows argon in the table. Farther on in the table there is a similar discrepancy. The row is as follows:—

Ag = 108, Cd = 112, In = 114, Sn = 119, Sb = 120.5,
Te = 127.7, I = 127.

The differences are:—

4.0, 2.0, 5.0, 1.5, 7.2, .07.

Here, again, there is a negative difference between tellurium and iodine. And this apparent discrepancy has led to many and careful re-determinations of the atomic weight of tellurium. Professor Brauner, indeed, has submitted tellurium to methodical fractionation, with no positive results. All the recent determinations of its atomic weight give practically the same number, 127.7.

Again, there have been almost innumerable attempts to reduce the differences between the atomic weights to regularity, by contriving some formula which will express the numbers which represent the atomic weights, with all their irregularities. Needless to say, such attempts have in no case been successful. Apparent success is always attained at the expense of accuracy, and the numbers reproduced are not those accepted as the true atomic weights. Such attempts, in my opinion, are futile. Still, the human mind does not rest contented in merely chronicling such an irregularity; it strives to understand why such an irregularity should exist. And, in connection with this, there are two matters which call for our consideration. These are:—Does some circumstance modify these "combining proportions" which we term "atomic weights"? And is there any reason to suppose that we can modify them at our will? Are they true "constants of Nature," unchangeable, and once for all determined? Or are they constant merely so long as other circumstances, a change in which would modify them, remain unchanged?

In order to understand the real scope of such questions, it is necessary to consider the relation of the "atomic weights" to other magnitudes, and especially to the important quantity termed "energy."

It is known that energy manifests itself under different forms, and that one form of energy is quantitatively convertible into another form, without loss. It is also known that each form of energy is expressible as the product of two factors, one of which has been termed the "intensity factor," and the other the "capacity factor." Professor Ostwald, in the last edition of his "Allgemeine Chemie," classifies some of these forms of energy as follows:—(see next column).

In each statement of factors the "capacity factor" is placed first, and the "intensity factor" second.

In considering the "capacity factors" it is noticeable that they may be divided into two classes. The two first kinds of energy, kinetic and linear, are *independent of the nature of the material* which is subject to the energy. A mass of lead offers as much resistance to a given force, or, in other words, possesses as great inertia, as an equal mass of hydrogen. A mass of iridium, the densest solid,

Kinetic energy is the product of Mass into the square of velocity.

Linear	"	"	Length into force.
Surface	"	"	Surface into surface tension.
Volume	"	"	Volume into pressure.
Heat	"	"	Heat-capacity (entropy) into temperature.
Electrical	"	"	Electric capacity into potential.
Chemical	"	"	"Atomic weight" into affinity.

counterbalances an equal mass of lithium, the lightest known solid. On the other hand, surface energy deals with molecules, and not with masses. So does volume energy. The volume energy of 2 grms. of hydrogen, contained in a vessel of 1 litre capacity, is equal to that of 32 grms. of oxygen at the same temperature, and contained in a vessel of equal size. Equal masses of tin and lead have not equal capacity for heat; but 119 grms. of tin has the same capacity as 207 grms. of lead,—that is, equal atomic masses have the same heat capacity. The quantity of electricity conveyed through an electrolyte under equal difference of potential is proportional, not to the mass of the dissolved body, but to its equivalent,—that is, to some simple fraction of its atomic weight. And the capacity factor of chemical energy is the atomic weight of the substance subjected to the energy. We see, therefore, that while mass or inertia are important adjuncts of kinetic and linear energies, all other kinds of energy are connected with atomic weights, either directly or indirectly.

Such considerations draw attention to the fact that quantity of matter (assuming that there exists such a carrier of properties as we term "matter") need not necessarily be measured by its inertia, or by gravitational attraction. In fact, the word "mass" has two totally distinct significations. Because we adopt the convention to measure quantity of matter by its mass, the word "mass" has come to denote "quantity of matter." But it is open to anyone to measure a quantity of matter by any other of its energy factors. I may, if I choose, state that those quantities of matter which possess equal capacities for heat are equal; or that "equal numbers of atoms" represent equal quantities of matter. Indeed, we regard the value of material as due rather to what it can do, than to its mass; and we buy food, in the main, on an atomic, or perhaps a molecular, basis, according to its content of albumen. And most articles depend for their value on the amount of food required by the producer or the manufacturer.

The various forms of energy may therefore be classified as those which can be referred to an "atomic" factor, and those which possess a "mass" factor. The former are in the majority. And the Periodic Law is the bridge between them; as yet, an imperfect connection. For the atomic factors, arranged in the order of their masses, display only a partial regularity. It is undoubtedly one of the main problems of physics and chemistry to solve this mystery. What the solution will be is beyond my power of prophecy; whether it is to be found in the influence of some circumstance on the atomic weights, hitherto regarded as among the most certain "constants of Nature"; or whether it will turn out that mass and gravitational attraction are influenced by temperature, or by electrical charge, I cannot tell. But that some means will ultimately be found of reconciling these apparent discrepancies, I firmly believe. Such a reconciliation is necessary, whatever view be taken of the nature of the universe and of its mode of action; whatever units we may choose to regard as fundamental among those which lie at our disposal.

In this address I have endeavoured to fulfil my promise to combine a little history, a little actuality, and a little

prophecy. The history belongs to the Old World; I have endeavoured to share passing events with the New; and I will ask you to join with me in the hope that much of the prophecy may meet with its fulfilment on this side of the Ocean.

A POSSIBLE NEW ELEMENT, OR POSSIBLE NEW ELEMENTS, IN CAST-IRON AND BLAST-FURNACE BOILER-DUST.

By G. G. BOUCHER.

It will appear at first somewhat surprising to those accustomed to make analyses of cast-iron that there exists in that metal another element besides the elements C, Si, S, P, Mn, Cu, As, Sb, Cr, W, Ti, Ni, Co, Al, K, Na, Mg, Ca, Li, and V, already known to be present in cast-iron. There does, however, appear to be another metal present, and in this article I have attempted to describe a metal which I discovered in the iron and boiler-dust made at these works. I have described it as a possible new element, because as yet it has not been examined spectroscopically, and, although its properties are unlike those of any metal I know, it is possible that it may be one of those compounds so difficult to separate by chemical means, the properties of which can only be determined by means of the spectroscope. I thought it better, therefore, not to call it a new element till the result of the spectrum analysis is known. A small quantity of the metal is now in the hands of Sir William Crookes, to whom I wrote describing my discovery, and requesting that he would take a little for analysis. He very kindly consented to do so, and I hope in a short time to be able to give the result of his analysis. In the meantime I have written this article hoping that it may prove interesting.

To prepare the metal it is necessary to take large quantities of iron, as the metal is present in very small quantities. On four different occasions I obtained 0.0033 per cent, 0.0027 per cent, 0.0053 per cent, and 0.0060 per cent of the metal, using 100 grms. of iron in each case. An analysis of an average sample of iron ore only yielded 0.0019 per cent of the metal.

To obtain the metal, 100 grms. of iron are weighed out into a large beaker capable of holding about 3 litres; 2000 c.c. of dilute H_2SO_4 (1 acid to 5 H_2O) are run in, the beaker is covered with a clock glass, placed on a warm plate, and the iron completely dissolved. The beaker is then removed, the contents allowed to cool, H_2S is passed through the solution to precipitate any small quantities of metal which may have dissolved, and it is then allowed to stand till all the graphite, &c., have settled. The clear liquid is poured off; the graphite, &c., are filtered into a quick filter-paper, and thoroughly washed with hot water till free from iron. The graphite, SiO_2 , As, Sb, Cu, and unknown metal collected on the paper, are washed off into a beaker and treated with 4 or 5 grms. of $KClO_3$ and 50 c.c. HCl . The beaker is placed on a hot plate and gradually heated to boiling, and boiled till all free Cl is evolved. The metals being converted into chlorides, the silica and graphite are filtered off, well washed with hot water, and the filtrate saturated with H_2S . The precipitate—which consists of the sulphides of As, Sb, Cu, and unknown metal—is allowed to settle, then filtered, and well washed with H_2S water. The sulphides with filter-paper are placed in a small beaker, HCl is added, and about 2 grms. $KClO_3$; the beaker is placed on a warm plate, and when the sulphides are completely dissolved and the free chlorine driven off, the solution is filtered, the bulk of the liquid being kept as small as possible. The As is precipitated by $MgCl_2$, $AmHO$, and $AmCl$, the solution being allowed to stand with repeated stirring about twelve hours. When the As is entirely precipitated it is filtered off, and the solution saturated with H_2S to

precipitate the Cu; the CuS is allowed to settle, filtered off, and the solution made slightly acid with HCl . The Sb and unknown metal are precipitated as sulphides, the precipitate being as a rule of a brown colour. The sulphides are allowed to settle, and when the liquid is quite clear it is poured off; 30 c.c. HCl (1 acid to 2 H_2O) are poured on to the sulphide, and the solution boiled. The precipitate is allowed to settle, the clear liquid poured off, and another 30 c.c. dilute HCl are added; the solution is boiled again, and the precipitate then allowed to settle. This is repeated three or four times, till all the Sb_2S_3 is completely dissolved. The unknown metal remains behind as a heavy dark brown sulphide, it being almost insoluble in boiling dilute HCl (1 to 1). This sulphide is thoroughly washed with hot water, and then dissolved in a dilute solution of KHO ; the solution is saturated with H_2S , and filtered from any small quantities of CuS or other sulphides formed. The filtrate is made slightly acid with HCl and the brown sulphide precipitated filtered off; it is again dissolved in KHO , the solution saturated with H_2S , and filtered. HCl is added to precipitate the sulphide; it is filtered on to a paper, and thoroughly washed with hot water. This process is repeated till the KHO solution of the sulphide produces no precipitate with H_2S . The brown metallic sulphide—which should now be absolutely free from As, Sb, Sn, Bi, and Cu—is thrown on to a filter-paper, thoroughly washed with hot distilled water, and dissolved in hot HNO_3 (1 to 2). The solution is filtered, and made just alkaline with $AmHO$ to precipitate, or make doubly sure of the absence of, Sb, Sn, Bi, and Te. These metals are, however, hardly likely to be present. The metal is again precipitated as sulphide, filtered off, well washed with hot water, and dried in the water-oven. When quite dry the precipitate is brushed off the filter-paper into a porcelain crucible, and placed in a muffle which is hardly red-hot. The sulphide burns to an oxide of a pale yellow colour, which melts very easily, and at a full red-heat completely vapourises; hence it is necessary to keep the crucible at a very low red-heat, and to watch this part of the process very carefully. The oxide can also be obtained by dissolving the sulphide in HNO_3 , evaporating to dryness, and heating gently as described before, but it is not in such a good condition for reduction as that obtained by oxidation in a muffle. The metal can be obtained from the oxide either by heating it in a current of hydrogen or by fusing it with pure KCN .

The metal so obtained is a black powder, slightly soluble in cold strong HCl and H_2SO_4 , and very little more soluble on boiling in these acids. It is soluble in dilute and strong HNO_3 , and very easily soluble in aqua regia. It is insoluble in dilute HCl and H_2SO_4 .

Heated in a current of air the metal glows, and is converted into the yellow oxide, part of which vapourises and condenses on the side of the tube.

The oxide so formed, as I have said before, melts at a low temperature, and when allowed to cool crystallises out in long transparent pointed crystals, which are very beautiful when examined through the microscope. If a small quantity of the oxide be placed in a covered porcelain crucible and heated to a full red heat for one or two minutes in a muffle, on taking it out and examining it the crucible side will be found to be covered with long transparent colourless crystals. I have in my possession crystals prepared in this manner over a quarter of an inch long. The first lot of crystals I obtained were very nearly half an inch long and almost completely filled the crucible; they were quite transparent and appeared to be capable of refracting light.

The oxide, heated with borax, gives in the outer flame a clear colourless bead, and in the reducing flame a light-coloured pink bead. Heated with microcosmic salt it gave a clear chrome-green bead, both in oxidising and reducing flame; the bead obtained in the reducing flame being considerably darker in colour than that obtained in the oxidising flame. Fused with Na_2CO_3 it gave a colourless mass completely soluble in water. The oxide is difficultly

soluble in HCl, almost insoluble in H_2SO_4 , and insoluble in HNO_3 .

WET REACTIONS.
Chloride of the Metal.

Reagent.	Observation.
HCl	No precipitate.
H ₂ S	A dark brown precipitate from slightly acid solutions, soluble in SAm_2 , S_2Am_2 , Na_2S , KHO, and NaHO; insoluble in boiling dilute HCl (1 to 1) and H_2SO_4 ; soluble in aqua regia and HNO_3 .
NaHO, KHO, AmHO	No precipitate, but slight blue colouration of the solution.
$Na_2S_2O_3$	A violet colouration, which turns brown on heating with a few drops of HCl; the metal being precipitated as sulphide.
Na_2SO_3	No precipitate or apparent change even on boiling.
$SnCl_2$	No precipitate or apparent change even on boiling.
$FeSO_4$	No precipitate or apparent change even on boiling.
$BaCO_3$, (Am) $_2CO_3$..	No precipitate.
$CaCO_3$, Na_2CO_3 ..	" "
Magnesia mixture (AmCl and AmHO)	" "
AmCl, KCl	" "
Zn, Fe	A fine black deposit of metal. Part of the metal is evolved in combination with hydrogen. On lighting the issuing gas at a jet and placing a cold porcelain lid in the flame, the metal is deposited. The deposit is almost black and has very little lustre. It is insoluble in HCl, and a freshly prepared solution of bleaching powder has no action on it.
K_2CrO_4	No precipitate.
KCN	" "
Na_2HPO_4 and $NaC_2H_3O_2$..	" "
K_3FeCy_6	" "
K_4FeCy_6	A dark brown flocculent precipitate from neutral solutions. Soluble in acids and alkalis.
H_2SO_4 , HCl	The most characteristic reaction for this metal appears to be that produced by the addition of a few drops of H_2SO_4 to the chloride or nitrate. On evaporating down till fumes of H_2SO_4 are driven off, and allowing to cool, the liquid assumes after a short time a most magnificent blue colour. Very minute quantities of the metal can be detected by this means. The same colour is produced by evaporating down two or three times with HCl to very nearly dryness. The colour is entirely destroyed by the addition of water.

The Metal in Boiler Dust.

A similar metal in every respect but one I have also found in the boiler dust. It has the same appearance, forms an oxide to all appearances the same as the other, and produces the same chemical changes with the reagents given with the exception of $SnCl_2$. This reagent produces a dark blue colour, which on boiling with a little HCl turns to a dark brown.

It is almost more difficult to obtain this metal from

boiler dust than it is to obtain the metal from iron, as it is present in such minute quantities. To obtain about 0.3 grm. of the metal I had to treat quite a ton of dust, and as this was done in small quantities, taking about 28 lbs. at a time, it required considerable time, patience, and labour. It is not always possible to obtain the metal, as at times it seems to disappear altogether; at one time I was unable to find it for quite two months. It also seems to be present only in the dust from a few of the boilers. The lighter dust does not appear to contain any of the metal, it being only in heavy and dark coloured dust that I have been able to find it.

To prepare the metal, about 28 lbs. of dust are placed in a large canvas filter and well washed with water, the washings being allowed to run into a Winchester quart bottle. To save using a large quantity of water, the washings are run through several times till they are saturated with the salts K_2SO_4 and Na_2SO_4 , which are present in large quantities. If the metal is present the solution generally has a dark yellow colour. H_2S is passed into this solution for about ten or fifteen minutes, and then about 50 c.c. strong HCl are run in and the bottle thoroughly shaken. The metal is precipitated as sulphide, and in about twelve hours settles at the bottom of the bottle. The clear liquid is syphoned off, and the sulphide washed into a large stoppered bottle, there to wait till many more such precipitates have been added to it.

When a sufficient quantity has been collected, it is filtered off, washed into a large porcelain basin, and dissolved in aqua regia. 15 c.c. H_2SO_4 are added, and the solution is evaporated down till fumes of H_2SO_4 appear. The solution is allowed to cool, water is added, and it is then filtered from the insoluble residue of $PbSO_4$, SiO_2 , S, &c. The filtrate is made alkaline with AmHO and saturated with H_2S . The CuS , Bi_2S_3 , &c., are filtered off, washed with SAm_2 water, and the solution is acidified with HCl to precipitate the metal. It is filtered on to a paper, well washed with hot water, afterwards dissolved in KHO, and treated, as described before, under the preparation of the metal from iron.

A glance at the reactions of these metals will show that there is some reason to believe that there is something new about them—as will be easily seen from a comparison of the reactions of these metals and those which they seem to most closely resemble. It is more than probable that these metals will turn out to be the same, but whether spectrum analysis will show them to be metals already discovered remains to be seen. I hardly think they can be. These metals are, however, very interesting, inasmuch as I believe they have not been discovered in cast-iron or boiler dust before.

It may be interesting to know that I also found in the boiler dust the metals thallium, cadmium, zinc, lead, bismuth, arsenic, and antimony.

North Lonsdale Iron and Steel Co.,
Ulverston, August 18, 1897.

NITROGEN IN ANALYSES.*

By C. F. JURITZ, M.A.,
Senior Analyst, Government Laboratory, Cape Town.

THE ordinary farmer is no doubt much perplexed at the wide range of figures expressing the percentages of nitrogenous constituents, when comparing the results of analyses by different authors, of articles regarding whose composition he is desirous of learning something.

Comparing one fertiliser with another of the same class, such as bone meal, for example, one authority may tell him that it contains 34 per cent of nitrogen, another that it contains 20 per cent of nitrogenous matter, while

* Abridged from the *Agricultural Journal*, Cape of Good Hope, June 10, 1897.

a third might possibly return it as containing $4\frac{1}{2}$ per cent of ammonia.

Suppose a farmer is using linseed cake, and wishes to compare results. He finds one cake contains 27 per cent of albuminous compounds, while another has only $4\frac{1}{2}$ per cent of nitrogen. "What a difference!" he says, thinking that the same substance is referred to in each case. Again, he finds that wheat contains 12 per cent of nitrogenous compounds, while peas do not contain more than 4 per cent of nitrogen; this puzzles him, because he has always understood that peas were of the "nitrogen-loving" sort.

It is really a very simple matter to reconcile these seeming differences. As 17 parts of ammonia contain 14 parts of nitrogen, we know that 17 per cent of ammonia is equivalent to 14 per cent of nitrogen. Again, the terms "nitrogenous matter," "albumenoids," &c., are frequently used interchangeably for a certain class of substances which contain about 16 per cent of nitrogen; that is, 1 part of nitrogen in $6\frac{1}{2}$ parts of nitrogenous matter. If, therefore, we have a bone-meal containing $3\frac{1}{2}$ per cent of nitrogen; to find how much ammonia this is equal to, we must multiply by 17 and divide by 14. The amount of nitrogenous matter is found by multiplying the nitrogen by $6\frac{1}{2}$ and *vice versa*.

Now, taking the first instance quoted, viz., the three samples of bone-meal—The first contained $3\frac{1}{2}$ per cent of nitrogen; the second 20 per cent of nitrogenous matter—that is, $\frac{20 \times 16}{100} = 3\cdot2$ per cent of nitrogen—or really

less than No. 1. The third shows $4\frac{1}{2}$ per cent of ammonia, or $\frac{4\frac{1}{2} \times 14}{17} = 3\cdot5$ per cent of nitrogen. Similarly

with the two samples of linseed cake; the latter contains more nitrogen than the former, being $4\frac{1}{2} \times 6\frac{1}{2}$, or practically 30 per cent.

As a matter of fact, most text-books explain their results by a footnote, as in the following analysis of hay:—

Moisture	16·50	per cent
*Nitrogenous substances ..	15·81	"
Carbonaceous principles ..	37·63	"
Woody fibre	22·47	"
Mineral matter	6·59	"
	99·00	

* Containing nitrogen, 2·53 (equal to ammonia, 2·08 per cent).

EXPERIMENTAL RESEARCHES ON GLASSES,

CARRIED OUT UNDER THE DIRECTION OF THE
"COMMITTEE OF CHEMICAL ARTS," OF THE SOCIÉTÉ
DE L'ENCOURAGEMENT.*

By L. GRENET.

THE object of these researches, for which the proprietor of the glass-works at Blanzay, M. Solvay, and the Company of Saint-Gobain, kindly offered the use of their works to the Committee, was to determine the relations existing between the chemical composition of glass and its most important properties, such as dilatation, fusibility, tenacity, refrangibility, and alterability. The first part, which concerns dilatation, and has been done in conjunction with M. Chatenet, is now finished, and forms the subject of this article.

Very little work has till now been done on the dilatation of glass—that of Fizeau, Schott, and Damour being all that is worth recalling.

The tension of an enamel applied to a body less dilatant is easily found by the following equation:—

$$1 - \Delta(T - t) = [1 - \delta(T - t)] \left(\frac{R}{E} + 1 \right)$$

* Abridged from the *Bull. de la Soc. de l'Encouragement*, Series 5, Vol. II., No. 6, June, 1897.

when Δ = the coefficient of dilatation of the body to be enamelled;

δ . Coefficient of dilatation of the enamel;

T. The temperature beyond which the enamel must not be raised without becoming permanently deformed;

t. The lowest temperature to which the enamelled body is reduced;

R. Tension of the enamel;

E. Coefficient of elasticity of the enamel.

Compared with metals the dilatation of enamel is almost always too feeble, and if the tension due to the difference of dilatation is too great the adherence of the enamel to the metal may be insufficient to resist it, and the enamel will crack off; but it is better, if the coefficients of dilatation cannot be made to agree, that that of the enamel should be the lesser, so that it remains under compression.

The measurements in all these experiments were made by M. Fizeau's method. The apparatus used was very completely described by M. Damour, modified by M. le Chatelier, in the *Bulletin* of February, 1896: the only important modification since this time has been the substitution of warming by means of water instead of by air. It consists of three points of tempered steel, which serve as a support for the glass under examination. Parallel to the base of this tripod, and 2 c.m. from it, are three levelling screws of hardened steel. The glass, cut into a prism of 2 or 3 c.m. high and polished on its lower surface, is placed on the tripod, and adjusted by means of the screws in such a manner that its polished face coincides almost exactly with the base of the tripod.

Under these conditions, if we place this over a biconvex lens, and illuminate with monochromatic light, we can observe Newton's rings. The prism and its support on the biconvex lens are placed on the top of a vertical tube, at the lower end of which two prisms are arranged, at its junction with a horizontal tube. The yellow light from a Bunsen is received on and totally reflected up by a prism, to the glass under experiment; from there it is reflected down the vertical tube again on to another prism fitted with a lens which again reflects it further along the horizontal tube, where the image of the rings can be examined on a screen by aid of a microscope.

On warming up the vessel containing the prism under examination and its support, the rings at first appear very rapidly, and the black centres which appear are counted. After about three-quarters of an hour the equilibrium of temperature becomes established and the rings no longer pass, and the temperature is noted. Each passage of a black centre corresponds to a difference in elongation of half a wave-length between the prism and its support. The dilatation of the glass in question is then found by the equation—

$$\delta = \Delta \pm \frac{1472 f}{\theta} \times 10^{-8},$$

when Δ = Dilatation of the support;

f. The number of rings which pass;

θ . Difference between the extremes of temperature.

There may be a systematic error due to the graduation of the support, but this is reduced to a minimum by taking the mean of several experiments.

Most of the samples of glass used in this research were made in a Schloesing furnace in a platinum crucible, then cast into triangular prisms 30 to 40 m.m. long and of 8 to 12 m.m. width of face, in moulds of clay or wood charcoal; they were then annealed at a dull red-heat, and allowed to cool very slowly; when cool they were ground, and one end was polished. By using platinum crucibles there is no fear of any variation in the composition of the glass.

Our first endeavours were to try and verify the truth of the law connecting dilatation and chemical composition, which was admitted by Schott without sufficiently conclusive proofs; he thought that the dilatation of glass in

creased in a similar manner to density, by the simple addition of certain bodies. To decide this point we had to have recourse not to ordinary commercial glass of complex character, but to glass of very simple character prepared in the laboratory, and of which the constituent parts varied in proportions as widely apart as possible. After becoming convinced of the inexactitude of this law, we tried to find some precise qualitative indications of the manner in which variations in a given glass affected its dilatation.

The curves made for the comparison of the results give to dilatation a function of the volume, and that seems more reasonable than to imagine a relation between dilatation and the weight of the component parts of the glass.

Silicates of soda and potash are vitreous, from the state of NaO, SiO_2 and KO, SiO_2 up to SiO_2 ; but when more alkaline than $\text{NaO}, 2\text{SiO}_2$ and $\text{KO}, 3\text{SiO}_2$, they become too hygrometric to allow of practical measurement of the dilatation.

All the silicates of lithium we prepared became devitrified. Contrary to what occurs with alkaline silicates, the dilatation of silicate of lithium increases with the proportion of silica: it is true that the mixtures are not vitreous, so this increased dilatation is probably due to the presence of crystals of silica; the mean dilatation of quartz is, in fact, very high, viz., 1206×10^{-6} .

Borates of soda and lithia can be obtained in the vitreous state, from the condition of $\text{NaO}, 2\text{BO}_3$, and $\text{LiO}, 3\text{BO}_3$, up to BO_3 .

The only borate of zinc obtainable is $\text{ZnO}, 0.67\text{BO}_3$, and this becomes devitrified very rapidly into cubic crystals.

One per cent of boric acid is lost when heated to bright red for fifteen minutes, but this small loss does not interfere with the measurements of the easily fusible borates. The measurements made on a large number of samples of borates show that there is a very sharply defined minimum,—that the dilatation decreases with a slight increase in the proportion of the bases; it reaches the minimum, and then increases rapidly. This is an important fact, and shows by itself that Schott's law is incorrect; moreover, the same was observed with more complex samples of glass containing boric acid.

As much as 60 per cent of boric acid can be added to white glass, but beyond that the excess separates out, and even with this high proportion the glass is not really homogeneous. Bottle glass devitrifies when there is more than 15 per cent of boric acid present, but even under the microscope there is no trace of crystallisation.

From the results shown in the table it can be seen that the coefficient of dilatation of boric acid is about 350, and it is this figure that must be taken in considering the influence of small additions of boric acid in commercial glasses.

In certain cases oxide of lead is, like boric acid, capable of giving a minimum of dilatation; but the effect produced is never strongly marked, and does not appear to be general. By adding 6 per cent of oxide of lead, M. Damour increased the dilatation from 514 to 551. It can be added up to 60 per cent without devitrification in the case of bottle glass, but the glass is then no longer homogeneous.

Alumina, when in large proportion, has the effect of making the glass almost infusible: this necessarily limits its use. Oxide of lead seems to be the body which best dissolves alumina. The measurements made show that when increasing quantities of alumina are added a minimum of dilatation is reached, in the same way as with borate of lead. An interesting fact noticed is, that the separation of boric acid from PbO_3 , 3BO_3 , and from $3\text{ZnO}, 2\text{BO}_3$, is stopped by alumina, although when in the crucible these borates are as liquid as water. Finally, the addition of small quantities of alumina to glass has the effect of lowering the coefficient of dilatation; this effect is more marked the more acid the glass is.

Some of the conclusions to be drawn from this research are, that a large number of bodies, such as BO_3 , PbO , CaO , MnO , Al_2O_3 , &c., when added to the glass in small quantity, lower the dilatation first to a minimum, and afterwards increase it as the proportion added is raised. This has not been observed in the case of potash, soda, or silica glass; possibly it occurs with such a minute addition as not to be practicable. Nevertheless the double silicates of potash and soda have a lower dilatation than the corresponding simple silicates. Alumina, while greatly lowering the dilatation, allows us, on account of the special fixity it communicates to glass, to obtain very alkaline silicates of high dilatation, offering great resistance to water.

Fluoride of calcium, in spite of the high dilatation given by fluorine, only increases it very slightly, and when the proportion added becomes at all large the glass is completely devitrified. To sum up, the bodies studied can be divided into two classes:—1. Those which increase the dilatation, KO , NaO , LiO , CaO , P_2O_5 , 3CaO , CaF_2 , and cryolite. 2. Those which decrease the dilatation, BO_3 , SiO_2 , Al_2O_3 , PbO , SiO , ZnO , Fe_2O_3 , and the colouring oxides.

NOTE ON THE CHEMICAL COMPOSITION OF THE MINERAL RUTILE.

By B. HASSELBERG.

In my researches on the arc-spectrum of titanium I employed, as elsewhere stated (*Svenska Vetensk. Akad. Handl.*; also *Ap. J.*, v., 194—198, 1897), instead of the commercial metallic powder, a Norwegian specimen of the mineral rutile, mainly on account of the far greater steadiness of the arc thus formed. According to the hitherto published chemical analyses of this mineral (Dana, "Descriptive Mineralogy," fifth edition, New York, 1883, p. 160), I had no good reason to expect any foreign lines of importance other than those of iron, the more conspicuous of which would be present in any case on account of impurities in the carbons. However, upon examining the arc-spectrum of vanadium obtained from a specimen of this metal presented to Baron Nordenskiöld by Moissan, of Paris, I found, to my great surprise, that several of its strongest lines coincided exactly with faint lines in my titanium spectrum, thus indicating a very appreciable percentage of vanadium in the rutile analysed. This induced me to investigate more closely the spectra of other specimens of the mineral in question, particularly as I had the opportunity to select from among the rich collections of the Royal Mineral Cabinet varieties from different quarters of the world.

In the comparisons I have used only the part of the spectrum included between $\lambda 460$ and $\lambda 427$. This is sufficient, for in this region there is situated one of the most prominent groups of the whole vanadium spectrum, namely, the group $\lambda 441-438$, the presence of which in the spectrum of any rutile, even though feeble in intensity, would indicate indubitably the presence of a sensible percentage of the metal. In order to decide definitely concerning the coincidences, the above-named part of the vanadium spectrum was photographed upon the same plate with the same region of the spectra of the different rutiles, and on these plates the intensities of the rutile lines corresponding to vanadium were estimated on a scale in which 1 denotes the faintest, and 6 the strongest lines. A + or - after a number indicates the intensity of the line in question to be nearer to this number than to the next. Thus, 1+ denotes an intensity greater than 1, but not attaining 1.2 and so on. In this way the following table has been constructed, which contains the results of the investigation of twelve rutiles, namely:—

Norway ..	..	1. Rutile from Kragerøe.
		2. " " Langøe.
		3. " " Loftheshagen.

TABLE A.
Rutile from—

Vanadium		Kragerøe.	Langøe.	Lofthagen.	Käringbricka.	Tachowaja.	Miask.	Binnenthal.	Yrieix.	Freiberg.	Castilia.	Graves Mountain.	Arkansas.	Remarks.
λ	I													
4268.85	3	..	I+	..	I	I	I-	..	trace	I-	I	I+	trace	The word 'trace' indicates an intensity too feeble to be estimated.
71.80	3	..	..	..	I	..	..	..	..	..	..	..	..	
4330.15	3	..	I-	trace	I	trace	trace	..	..	trace	trace	I	..	
33.00	3	..	I	I	I	I	trace	..	..	trace	I-	I'2	..	
41.15	3	..	I+	I	I-	I	trace	..	trace	I-	I	I'2	trace	
53.05	3.4	I	I+	I+	I'2	I+	trace	..	trace	I-	I	I'2	trace	
79.42	4.5	2	3	2.3	3-	2.3	2-	trace	2	2	2.3	2.3	2-	
84.95	4.5	2	2.3	2+	2.3	2+	I'2	trace	2-	2-	2+	2+	I'2	
90.15	4.5	2	2	2	2+	2	I	..	I'2	I'2	2-	2	I+	
95.40	4.5	I	I'2	I'2	2	I'2	trace	..	I+	I	I+	2-	I	
4400.75	4	I'2	2-	I'2	2	2-	I	I+	I'2	I'2	2-	2-	I+	
06.85	4.5	..	I'2	I'2	2-	I'2	trace	..	I	I	I+	2-	I	
07.90	4.5	I'2	2-	2-	2	2-	I	trace	I'2	I'2	2-	2+	I+	
08.40	4	I'2	2-	2-	2	2-	I	..	I'2	I'2	2-	2+	I+	
08.65	4.5	I'2	2-	2-	2	2-	I	..	I'2	I'2	2-	2+	I+	
16.65	3	2	..	..	2	..	..	..	..	..	..	..	..	
38.03	3.4	..	I	I-	I	I	I	..	..	trace	trace	I	..	
41.90	3.4	I'2	2-	2-	2-	I'2	I'2	I+	..	I'2	I'2	2-	I	Ti
44.40	3.4	..	2-	2-	I'2	I'2	I'2	I'2	..	I'2	I'2	2-	I	Ti
52.12	4	..	I	I-	..	I-	trace	trace	..	trace	I-	I'2	..	
59.95	4	trace	I+	I+	I'2	I'2	I-	..	I-	I	I	I+	I	
60.45	4.5	I	I'2	I'2	2-	2	I	..	I+	I'2	I'2	2-	I-	
62.55	3.4	trace	..	..	I	I	..	..	trace	trace	..	I	I	
69.90	3.4	..	..	..	I-	I-	trace	..	trace	trace	..	I-	I	
4545.62	3.4	..	..	..	I'2	I+	I	I	I	I	I	I+	I+	
49.85	3	3.4	..	..	3.4	3.4	3.4	..	3.4	3	3.4	3.4	3.4	Belongs to Ti, Co.
77.40	4	trace	..	..	I+	I+	I-	..	trace	I	I	I	trace	
80.55	4	I	..	..	I'2	I'2	I	..	I	I+	I	I+	I	
86.55	4.5	I	..	..	I'2	I'2	I-	..	I	I+	I	I+	I	
94.30	4.5	I	..	..	I'2	2-	I-	..	I	I+	I	I+	I	

TABLE B.

Chromium.													
4254.49	6	trace	3	3	2.3	3	trace	..	2.3	2.3	2.3	2.3	..
74.91	6	..	2.3	2.3	2-	2+	..	..	2	2	2	2+	..
89.87	6	..	2	2	2-	2	..	..	I'2	2-	2-	2	..

Sweden	4.	Rutile from Käringbricka.
Russia	5.	" " Tachowaja, Ural.
	6.	" " Miask, Orenberg.
Switzerland ..	7.	" " Binnenthal, Wallis.
France	8.	" " Yrieix.
Germany	9.	" " Freiberg.
Spain	10.	" " New Castilia.
America	11.	" " Graves Mountain, Lincoln Co.
	12.	" " Magnet Caves, Arkansas

From Table A it will be seen that, with one exception, all the rutiles examined contain vanadium in varying proportions. This exception is found in the Anatas from Binnenthal, Canton Wallis, in Switzerland, in the spectrum of which the vanadium lines are almost absolutely wanting. This statement is not invalidated by the greater intensity of the two lines 4444.40 and 4441.90, for these lines belong without doubt to titanium, although they differ so very little in position from the vanadium lines that a separation on my spectrograms is impossible.

On comparing the intensities of the vanadium lines in the different specimens of rutile, the singular fact at once manifests itself that varieties from neighbouring lodes contain a very different percentage of the metal. Thus, among the Norwegian rutiles, the two specimens from Langøe and Lofthagen contain vanadium in a much larger proportion than the Kragerøe rutile, and the same holds good for the two Russian and also for the American rutiles. This peculiarity finds a counterpart in the case of another component of some rutiles, namely, chromium, of which metal a very notable amount was discovered in the Swedish specimen from Käringbricka as early as 1803

(Dana, "Mineralogy," p. 161), and is now detected in some of the varieties under discussion.

In order to prove that the observed titanium lines are not to be ascribed to an impurity of the carbons, the spectrum of the latter was photographed with that of vanadium before introducing rutile into the arc. Besides the ordinary carbon bands the resulting plates show feebly only a few of the most conspicuous iron and calcium lines, but of vanadium not the least trace is seen. The purity of the carbons analysed is thus to be considered as entirely satisfactory.

While the presence of vanadium in the rutile thus forms a hitherto entirely unknown feature of this mineral, the presence of chromium in the Swedish variety was, as above stated, detected by chemical analysis as early as 1803, and has since then been verified in some other specimens. The present method of research, however, permits of a much easier decision in this respect on account of the occurrence of one of the strongest groups of the whole spectrum of chromium, viz., λ 4289.9, 4274.9, 4254.5 just within the part here photographed. It is not very difficult to find them out among the crowd of titanium lines on the photographs, and from their estimated intensities, to form at least an approximate idea of the greater or less quantity of chromium contained in the specimens examined. The results of these comparisons are given in Table B.

It will be seen that in different rutiles the chromium lines show differences of intensity, fully justifying the conclusion of a corresponding disparity in the amounts of the metal. Thus, while the Anatas and also the Arkansas rutile are absolutely free from chromium, and in the rutiles from Kragerøe and Miask only a feeble trace is

present, the other specimens contain a very considerable percentage of this metal. But the most peculiar feature in this respect appears upon comparing chromium with vanadium. It is thus found that in those varieties of rutile which contain vanadium in any very appreciable amount, chromium is also present, while a small percentage of the former metal is accompanied by a corresponding scarcity or even complete absence of the latter.

In conclusion, it should be remarked that in the case of the Norwegian rutile from Langöe the preceding results have been completely confirmed by ordinary chemical analysis kindly undertaken by Baron Nordenskiöld. It is thus evident that the accepted chemical analyses of the present mineral by no means possesses the completeness or accuracy which the usual chemical methods are capable of giving.—*Astrophysical Journal*, vi., No. 1, June, 1897.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING JULY 31ST, 1897.

By SIR WILLIAM CROOKES, F.R.S.,

and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolis Water Act, 1871.

London, August 10th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 189 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from July 1st to July 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in a previous report.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 189 samples examined all were found to be clear, bright, and well filtered.

The rainfall at Oxford during July was 2.57 inches, of which 2.25 inches fell on the 19th, 20th, and 21st inst.; the average for the last 30 years is 2.63 inches; this leaves a deficiency of 0.06 inch on the month. There was an excess of 0.06 inch on the first six months of the year; so this is exactly balanced by the deficit of this month; and the actual fall this year, up to the end of July, is identical with the 30 years' average, viz., 13.94 inches.

The results of our bacteriological examinations of 265 samples are recorded in the following table; we have also examined 53 other samples taken at special points, stand-pipes, wells, &c., making a total of 318 samples:—

	Microbes per c.c.
Thames water, unfiltered (mean of 27 samples)	4971
Thames water, from the clear water wells of five Thames-derived supplies (mean of 133 samples)	83
Ditto ditto highest	496
Ditto ditto lowest	4
New River, unfiltered (mean of 27 samples) ..	754
New River, filtered (mean of 25 samples) ..	44
River Lea, unfiltered (mean of 27 samples) ..	2494
River Lea, from the clear water well of the East London Water Company (mean of 26 samples)	125

The average bacteriological quality of the London waters has been good during the month. On some days, however, in the case of two of the Thames-derived waters, there has been an excess of microbes, and the water from the Lea has not all the time been up to its best standard. We have been in communication with the Companies respecting certain alterations in the filter beds, which are likely to improve their efficiency. Our suggestions are being considered, and during the latter part of the month the bacterial quality of the water has greatly improved.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.
JAMES DEWAR.

PAINT TESTS.

MAX TOLTZ, in a paper read before the Civil Engineers' Society of St. Paul, December 7th, 1896, and printed in the *Journal of the Association of Engineering Societies* (June, 1897), has gone comprehensively into the comparative value of paints for protecting iron surfaces. The paints experimented with were (1) true asphaltic varnish paints; (2) so-called asphaltic varnishes, of inferior qualities; (3) black carbon paints, of which the vehicle is practically a varnish; (4) iron oxide paints; (5) graphite and silica graphite paints. Red-lead was not tested. Within the last ten years this material has been to a large extent discarded by progressive engineers, and although it has still warm advocates, even they are beginning to add carbon-black or graphite to it, says Mr. Toltz. One set of tests made by Mr. Toltz consisted in painting sheet-iron dishes, 12 inches diameter by 0.5 in. deep. The scale or skin was carefully removed before painting. Two dishes were then painted with each kind of paint, one receiving one coat and the other two coats, the first coat having dried at least a week before the second was put on. After the second coat had dried thoroughly, a given amount of water was placed in the dishes and allowed to evaporate at the ordinary temperature of the room, this being repeated until the dishes showed more or less rust. After most of the water had evaporated there remained at the junction around the edge a thin film of water, which in contact with the air, and the carbonic and other acids of the air, acted on the paint in such a way that the iron under it began to rust. In actual practice the same thing will happen, the only difference being that the rust will extend under the paint and will not show as plainly as on the dish. This is a severe test, but in Mr. Toltz's opinion no paint which fails to withstand it is desirable for the protection of iron and steel structures. The cheap asphaltum paints and iron oxide paints failed under this test. Another severe test consisted in exposing sheet iron coated with various kinds of paint to a temperature of 220–300° F., this test being of value as showing promptly whether a paint will keep its elasticity or will become so brittle that it may be easily removed from the surface.

In the discussion which followed this paper exception was taken to various statements made by Mr. Toltz, but the expressions of opinion were generally in support of his conclusions.

From our present knowledge, the following system for painting iron and steel bridges, and other metallic structures, is recommended by Mr. Toltz:—

First. Give the iron and steel a coat of the best grade of refined linseed oil, properly boiled and settled clear; or, still better, mix linseed oil with about 10 per cent of a good grade of lamp-black,—this coat to be applied at the mills, the iron or steel being first carefully cleaned from loose scales.

Second. After the structures have been erected, give

them one coat of real asphaltic varnish paint, made from the best grade of asphalt, linseed oil, and gum, compounded properly, so as to form a true varnish; or of a paint made from carbon black and properly boiled varnish, compounded of the best grade of linseed oil and gum. This coat should be carefully applied by a skilful painter, after the metal has been thoroughly cleaned from all loose scale, rust, shavings, filings, shrivelled oil or paint, grease, dirt, or any foreign matter, because it is of the utmost importance that the paint should be spread and worked in such a way so as to cover the surface properly, and be as free as possible from air-bubbles and form a continuous coating. This priming or first coat should be applied fairly thick, the thickness depending, to some extent, on the nature of the paint used. Before the second coat is applied, the first one should be thoroughly dried and hardened by natural oxidation, which will require at least ten days. If practicable, it would be a great deal better, as well as more economical, to apply the second coat not less than four weeks after the first one.

Third. As a second coat, a good grade of graphite paint is to be applied as thickly as possible, working the paint thoroughly with the brush. From the examinations made of the various grades of graphite paints, as far as graphitic pigments are concerned, there appears to be but little difference between them, provided, of course, that the pigment contains at least 33 per cent of pure graphite, the rest of the pigment being natural rock, ground very fine in pure linseed oil. The graphite paint should be bought in paste form, well ground, and contain not less than 70 per cent of pigment and 30 per cent, by weight, of the best quality of boiled linseed oil; the paste should be mixed with boiled linseed oil at the place where it is to be applied. No turpentine, no benzine, and no Japan or driers should, under any circumstances, be allowed in this paint.

Fourth. There are certain parts of steel or iron bridges, viaducts, or tunnels that should have an additional (third) coat of paint. These include such places, or parts of structures, as are directly exposed to the steam, fumes, and gases from passing engines. For such a coat some cheaper asphalt paints, applied very thickly over the coats above recommended, would be all-sufficient. Such a coat would protect the underlying primary coats for many years, preserving their natural toughness and elasticity, and preventing atmospheric action on the structure.

From the investigations made, as well as from practical experiments, it appears that the iron-oxide paints are not very desirable, at least for the first coat or two, for iron or steel; but as a third coat, for the protection of the underlying paints, they may be recommended.

However, the extensive investigation of the graphite paints that can be obtained in the markets to-day shows that, if properly applied, they are far superior to iron-oxide paints for the second or third coat, especially as they withstand the action of moisture and water much better than the best iron-oxide paint so far examined. Besides, a graphite paint, in paste form, well ground and mixed with boiled linseed oil, will not cost very much more per gallon than the cheapest iron-oxide paint in the market.

In recommending asphalt varnish paint or carbon paint for the first coat, great stress is laid upon the necessity of having the surfaces of iron or steel as free from moisture as possible while the structures are being painted, otherwise there is great danger that the coating will not adhere very firmly, and that it will thus actually nullify the value of the paint. This precaution is less important when an ordinary iron-oxide paint or red-lead paint, simply mixed with linseed oil, is used; because linseed oil itself has the property of absorbing moisture quite readily, whereas carbon or asphalt paint will not. The lack of this property in the two last-named paints is one of the principal reasons why they are superior to any other class of paints.

—*Engineering and Mining Journal.*

NOTICES OF BOOKS.

Twentieth Annual Report of the Connecticut Agricultural Experiment Station for 1896. Pp. 414. New Haven: The Tuttle, Morehouse, and Taylor Press. 1897.

THE Report on Food Stuffs, which comes first in this volume, contains the results of examinations of 849 articles of food, of thirteen different kinds. With the exception of Martius yellow, found in minute quantity in certain samples of mustard, no poisonous adulterants have been found, though 254 samples were found to be adulterated and 24 were reported as doubtful. The most frequent adulteration was found in coffee. White strained honey was, as a rule, the purest article examined.

The law with regard to adulteration seems to be in a very lax condition. It appears that a "Dairy Commissioner" was appointed under the Statute regulating the sale of *imitation butter, molasses, and vinegar*, but no one is charged with the execution of the laws regarding the adulteration of milk, candy, spirituous liquors, drugs, and medicines. Boards of Health, we are informed, are permitted to act under the Statute regarding the adulteration of food; but there is no record of any action having been taken under any of these Statutes. Surely the work of the "Dairy Commissioner" is somewhat anomalous.

Under the Report on Commercial Fertilisers we note that 255 distinct brands of fertilisers have been offered for sale by fifty-two firms. During the year 492 samples of fertilisers and manurial waste products have been analysed, results of the examinations being given in detail; after which comes a comprehensive review of the fertiliser market. The average monthly quotations show that nitrate of soda has ruled lower this year than for some years previously, and there has been but a slight demand for sulphate of ammonia in the Connecticut market. Nitrogen in the form of animal matter has also been cheaper.

A number of experiments have been carried out, and are here described, on various agricultural matters, such as the prevention of potato-scab, and the susceptibility of various roots to potato-scab, and it was found from trials made on eight different kinds of roots—viz., radishes, parsnips, salsify, carrots, turnips (two kinds), mangolds, and beets—that the radishes and carrots alone remained unquestionably free from scab, the salsify and parsnips showed little if any, while the turnips gave averages of 21 per cent and 15 per cent of scab, the mangolds 40 per cent, and the beets 63 per cent.

Other experiments dealt with a leaf blight of melons, a destructive fungus disease of tobacco, the so-called "shelling" of grapes, &c.

The work done at the Experimental Station is of varied character and of considerable interest, while great care is evidently exercised in carrying out the plan of campaign. The volume closes with a good Index of fifteen pages.

Contribution to the Polyhedral Origin of Species. ("Contribution à l'Origine Polyédrique des Espèces"). By ARTHUR SORIA ET MATA. Part I., pp. 203. Madrid: Chamartin de la Rosa. 1897.

THE author has studied the regular polyhedra, from the point of view of making them, and combinations of them, represent the elements and their compounds; and from the curious figures evolved he has endeavoured, with a certain amount of success, to reproduce the structure of chemical compounds and minerals, showing the angles, faces, planes of cleavage, &c., and he claims that if, from a mass of polyhedral groups, as shown by the figures he has built up, he can show a regular and systematic arrangement which reproduces a large number of—if not

all—the known crystalline forms, it must well be admitted that his hypothesis holds good until another and more perfect one comes to replace it.

Starting from the idea of an atom, which idea was not conceived without many doubts and much hesitation, we must admit motion, or no phenomena could take place; and from this point he works up to lines of atoms moving in unison and communicating their movement one to another, the total force being neither augmented or diminished, though passing and changing without ceasing from lines to faces, angles, and solid figures; this is not a long step, and he classifies all the figures necessary for his hypothesis into three groups, viz.:—the tetrahedron, the common origin of all bodies of three dimensions; the cube, which contains the beta-tetrahedron and the octahedron, the typical form of chemical species and minerals; and the dodecahedron, which contains many regular polyhedra, and represents the probable initial form of the cell, and all the animal and vegetable species. The theory is a pretty one, but is open to many objections,—still, as the author says, it may stand till another comes to replace it.

We cannot agree with the author's supposition of the interpenetrability of atoms; this supposition must concede mass, and therefore if two "masses" can penetrate each other "until their centres are coincident, without losing their respective independence of movement," there is an end of matter, as—carrying the argument further—all atoms would be self-contained in the mass, position, or volume, whichever it may be, of one,—which is absurd.

The book is accompanied by four large sheets of thick paper, on which are marked out a number of figures, all ready for cutting out and gluing up into the forms referred to in the letterpress.

OBITUARY.

VICTOR MEYER.

THE sudden and unexpected death of Professor Dr. Victor Meyer, on the 7th inst., has left a painful impression not merely at Heidelberg, but throughout the chemical world. The regretted event was at first ascribed to apoplexy, but it has since been stated—on what evidence it is not certainly known—that Prof. Meyer fell a victim to poisoning. We should add that those who regard the death of Prof. Meyer as suicide, allege that he suffered from domestic troubles.

Victor Meyer, the son of the calico-printer, Jacques Meyer, of Berlin, was born on September 8th, 1848. After his earlier education at the Werder Gymnasium he entered, in his sixteenth year, the University of Berlin, and after one term that of Heidelberg, where he devoted himself to the study of Chemistry under Prof. Bunsen, whose assistant he became. After graduating at Heidelberg he continued his studies at Berlin under A. von Baeyer. In 1871 he was called to the Polytechnicum at Stuttgart as First Assistant of Fehling, and (titular) Professor of Organic and Theoretical Chemistry. After a year he received a call as Professor of General Chemistry at the Polytechnicum at Zurich, *vice* Wislicenus. In 1885 removed to Göttingen, and in 1889 he was transferred to the University of Heidelberg as the successor of Bunsen, who had recommended him as the highly gifted of his pupils. Under his leadership chemical studies at Heidelberg experienced a rapid rise. In the last year from fifty to sixty applications had to be rejected in every term.

Through Victor Meyer's researches our science experienced important extensions in various directions. The methods of determining vapour densities were greatly simplified and facilitated by his procedures. His results at high temperatures (up to 1800° and beyond) yielded a firm basis for pyro-chemistry. We are indebted to him for

the discovery of the aldoximes and ketoximes (classes of bodies which become of fundamental importance for the recognition of the aldehyds and ketones). They became of prominent importance in the development of stereochemistry, especially that of nitrogen.

His most brilliant discovery in organic chemistry was that of thiophene in benzene, proceeding from which Victor Meyer may be said to have created a new branch of chemistry. As a Professor he was unsurpassed; few Academic teachers equalled him in the art of stimulating his hearers. Both his discourse and his carefully prepared experiments afforded his pupils a high enjoyment.

In 1885 Prof. Victor Meyer was elected an honorary member of the British Chemical Society.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 4, July 26, 1897.

Composition of Drainage Waters.—P. P. Dehérain. —From March, 1895, to March, 1896, the fields kept in fallow alone have yielded drainage water; the lands sown with annual plants, already impoverished in water by the vegetation itself, have been so thoroughly dried by the exceptional temperature of the autumn of 1895 that the autumnal rains could not saturate them. If we calculate for the surface of a hectare, we find that the drainage of the plots in fallow has removed 109 kilos. of nitric nitrogen—a figure analogous to that of 1893 and 1894, but very inferior to that of 1892. The rain has been very unequally distributed during the agricultural year, March, 1896, to March, 1897. Scanty at the outset, it became abundant in June, moderate in July and August, but it was extremely plentiful in September and October (130 and 136 m.m. respectively). The quantities of nitric nitrogen in the fallow land, deprived of nitrogenous manures, rose, during the moist years, to 200 kilos. per hectare, representing 1250 kilos. nitrate of soda, and exceed the requirements of the most greedy crops. The soils under crops elaborate a much smaller quantity of nitrates, for the abundant evaporation of the herbaceous plants dries the soil so completely as to admit of an energetic nitrification. When rain is very abundant we obtain, without nitrogenous manures, very good harvests, containing a sufficiency of nitric nitrogen.

Researches on the Conditions in which there occur Elements other than Carbon in Cast-Irons and Steels.—Ad. Carnot and M. Goutal.—This paper will be inserted in full.

Explanation of a Phenomenon attributed to a Magnetic Deviation of the X Rays.—Sir G. G. Stokes. —Already inserted.

Phthalic Green: its Preparation and Constitution.—A. Haller and A. Guyot.—Otto Fischer has given this name to a green pigment obtained in small quantities by causing phthalyl chloride to act upon dimethyl. The authors have found that in addition to diphenylphthalide there are formed small quantities of a new compound, $C_{20}H_{18}O$. This compound is formed by other methods, and in particular by the action of aluminium chloride upon phthalyl and benzene tetrachloride in the diphenyl-anthrone described by one of us in a former communication. The compounds here described are the hydrochlorate, $C_{32}H_{35}N_3OCl + H_2O$; the nitrate, $C_{32}H_{34}N_3O.NO_3$, is a very stable substance. The chloroplatinate, $(C_{32}H_{34}H_3OCl + 3HCl)_3PtCl_4$, is obtained in fine vermilion-red crystals. The leucobase of phthalic green has the composition $C_{32}H_{35}N_3O$.

Transformation of the X Rays by Metals.—G. Sagnac.—The various metals exert an elective absorption upon the X rays. At the same time the superficial layer of the metal emits anew rays much less easily transmitted than the X rays by mica, aluminium, black paper, and the air itself. These new rays are themselves transformed by aluminium. We foresee that we shall gradually succeed in filling up the space which separates the X rays from the known ultra-violet rays, and perhaps identify them with such rays.

Photographic Veiling in Radiography.—P. Villard.—Radiographic proofs often present a veiled aspect, especially in the case of thick objects. We admit readily that this result is due to X rays of a peculiar nature capable of traversing almost all bodies without notable absorption.

Addition of X Rays upon the Temperature of Animals.—L. Lecerclé.—Already inserted.

Line Spectrum of Carbon in Fused Salts.—A. de Gramont.—Tables of the wave-lengths forming the line-spectrum of carbon as recognised and measured in melted carbonates.

Relation between the Polymerisation of Liquids and their Dissociating Power upon Electrolytes.—Paul Dutoit and Mlle. E. Aston.

A New Group of Amidines.—Fernand Mutolet.—These two papers will be inserted as soon as possible.

Procedure for Determining Acetylene applicable to Carbides of the Form R—C——H.—M. Chavestelon.—The author brings back the determination of acetylene to an acidimetric estimation.

Determination of Lime, Alumina, and Iron in Mineral Phosphates.—L. Lindet.—This memoir will be inserted in full.

Absorption of Oxygen in the Fracture of Wines.—J. Laborde.

Bacteriological Study of Ambergris.—H. Beauregard.—Already inserted.

Revue Générale des Sciences Pures et Appliquées.
No. 14, July 30, 1897.

Annual Review of Pure Chemistry.—A. Etard.—One of the most important events of the year in pure chemistry is the liquefaction of fluorine by Messrs. Moissan and Dewar, whose paper has already been printed in full in these columns. Of the other less important work, the author sees nothing but a little more confusion, owing to the monotonous continuity of scientific productions of unequal value. The science of chemistry is extending every year in new directions. It now includes pressure, cold, electricity, &c., &c. It is being enriched principally by the determination of volumes, by the measurement of constants, and in more minute work striving to show if what we have so long admitted is really true. After a long period, during which it has seemed that science was getting so pure as to be useless, savants are coming back to the traditions of Gay-Lussac and Berthollet, and are endeavouring to adapt the modern lofty ideas to the wants of mankind. Thus, Lord Rayleigh proposes to make the direct oxidation of the nitrogen of the air commercially practicable by means of the electric currents which are now at the disposal of chemists. He found that with an electromotive force of 6000 volts the absorption of air in a 7-litre flask, in which played a spray of potash, reached 6880 c.c. per hour. Mr. Liversidge has tested various deposits of maritime origin for gold, and has found that the Stassfurt salts contain 0.13 grm. per ton. But it is not only gold that is so widely disseminated, but, as Mr. Hartley has shown, also the so-called rare earths and metals. In 92 different iron ores he found always either silver, rubidium, copper, gallium, indium, or thallium. Mr. Shenstone has found that the halogens Cl, Br, and I combine with mercury when in a

state of perfect dryness, but ozone will not do so except in the presence of aqueous vapour. Mr. Chattaway has studied the composition of iodide of nitrogen, and comes to the conclusion that NH_3I_2 is the most probable formula, certainly the rapport N : I₂ has been established.

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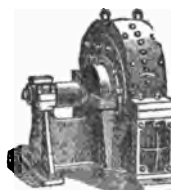
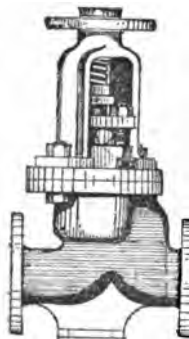
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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1971.

LABORATORY NOTES FROM NEW ZEALAND.

By WILLIAM SKEY,
Analyst to the Mines Department, N.Z.

1. If gold in potassic cyanide (weak or strong) is connected with platinum in an acid (say hydrochloric acid) and inter-polar connection made, a stream of hydrogen is given off from the platinum. For this experiment it is necessary to have the negative pole very small as compared with the size of the gold. I do not get this reaction if the platinum is in an alkaline solution,—probably because there is an oxidation of the hydrogen by the free oxygen in solution, induced by the affinity of potash for water. Copper is easily precipitated upon a gold or silver pole out of its sulphate, by gold or silver in a cyanide solution.

2. If platinum or gold, in a solution of tannic acid and potash, is paired with platinum or gold in an acid such as acetic or sulphuric acid, an evolution of hydrogen also takes place, and continues till all the tannic acid is oxidised. By the use of the tannic acid or tannin battery of a few cells, we can get all the manifestations of a very intense current. To conserve the tannic acid there should be a layer of kerosene oil upon it, to keep out the air; or the cells may be closed with cork, and the connections run through it.

In these two experiments it appears that, in the one case, it is the hydrocyanic acid of the hydrocyanate of potash that is decomposed, because this compound must be in the nature of things the most easily decomposable electrolyte present under the circumstances. Nature's work is always done, as we know, upon the lines of *least resistance*. In the second experiment it appears that water is decomposed. In neither of these experiments is it at all likely that any assistance is given to the operation by the electric current generated. The metals and the electrolytes merely allow of electricity being made, the free flow of which is necessary or favourable to continuous chemical action.

3. Platina that has been for three days in a solution of potash is still positive in that solution to platina in acids generally, and throws down gold on platina from its chloride or potassic aurate, also silver from its ammoniate. The platina was the purest I could obtain, and was digested for a long time with nitric acid, then well washed before use. An ignition of the metal prior to use did not affect the results.

4. Platina in a concentrated solution of common salt alkali-sed with potash that had been just boiled to expel air; also deposited gold from its chloride on a platina wire. As paired with platina in muriatic acid, it vigorously deflected a galvanometric needle for a short time; then deflected it feebly, but persistently, for an indefinite time.

The vigorous deflection may denote an oxidation of the platina itself, while the after deflection may denote an induced combination of the small minute quantity of oxygen and nitrogen that find their way to the surface of the platina that is in the saline solution. This has yet to be tested.

5. Platina in a weak or strong potash solution, paired with platina in a solution of ferrosulphate, is positive thereto, and if a decomposition cell with platina poles and an electrolyte of auric chloride be put in the circuit, a little gold is soon deposited upon that pole which is metallically connected with the platina in the potash solu-

tion. These results clearly show that a chemical action is taking place in the potash cell that is of a more intense character than that which we have in the case of oxidation of protoxide of iron to the sesquioxide.

For these kind of experiments bibulous paper for the electrical connection is not at all adapted, as the mixing of the electrolyte has to be avoided. The best method that I have yet devised is the "*gelatine connection*." A strong aqueous solution of gelatine is poured into a glass tube bent to a convenient form till it is full. This when cold is very convenient for the purpose, and absolutely prevents the blending of the solutions for days or weeks, according to their nature. To increase their conductive power where necessary, a little salt may be used. When made very stiff, even, it still conducts, showing that the molecules therein are still capable of moving to form the inter-polar lines.

6. If an insulated voltaic cell be connected with the insulated silver plates—say 6 inches square and about an inch apart—in the air, a current of electricity still obtains; this is so weak, however, that a pretty sensitive galvanometer does not indicate it. By passing the current into a solution of auric chloride by means of platina poles gold is deposited in a day or so (on the positive pole) in a very thin, coherent, continuous, and bright film. With the positive pole of gold the action is much accelerated.

RESEARCHES ON THE STATE IN WHICH ELEMENTS OTHER THAN CARBON ARE FOUND IN CASTINGS OF STEELS,

By AD. CARNOT and M. GOUTAL.

We propose here continuing the study of the chemical condition of the elements which enter into the composition of castings and steels, occupying ourselves first with the metals properly called manganese, copper, nickel, and chromium; then the rarer elements generally ranged along with the metals titanium, tungsten, and molybdenum.

Manganese.—The experiments which we have already described show that manganese has an especial affinity for sulphur and silica, and that, when it is in small proportion in a casting, it may be found entirely in the state of manganese sulphide or silicide. When it is in a large proportion, the solvents which we employ for iron cause the manganese to disappear at the same time.

Copper.—Potassium cupro-chloride does not allow us to isolate the copper contained in a steel; but we may effect this easily by the use of a weak acid, such as hydrochloric acid at 5 per cent, if it is employed with the exclusion of air, e.g., in a current of carbonic acid gas.

Nickel.—Nickel disappears entirely under the action of the neutral potassium cupro-reagent.

Chromium.—Ferro-chromes of a high standard are not readily attacked by acids. Hence we have been obliged to operate upon steels containing not more than 2.50 per cent of chrome. The insoluble residues consist of chrome, iron, and carbon.

Titanium.—The ferro-titanates may be attacked either by acids or by the cupric salt. The titanium is uncombined.

Tungsten.—The attack of a tungsten steel by dilute hydrochloric acid, at a gentle heat and with the exclusion of air, leaves as residue a compound of iron and tungsten of the composition Fe_3W .

Molybdenum.—Molybdenum steels, treated with dilute acids in the absence of air, leave a residue answering exactly to the Fe_3Mo_2 .

In fine, manganese, nickel, copper, and titanium seem to be simply dissolved in the steels; a portion of manganese may be in the state of sulphide or silicide in the cast metals,

Chromium forms complex and perhaps multiple compounds with iron and carbon.

Tungsten and molybdenum are in the state of definite combinations with iron represented by the formulæ Fe_3W and Fe_3Mo_2 .

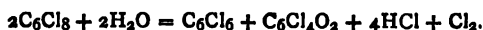
These elements, generally considered as metals, behave therefore in steel like non-metals, whilst arsenic, on the contrary, plays a part analogous to that of the true metals.—*Comptes Rendus*, cxxv., p. 221.

ON A COLORIMETRIC REACTION OF DISULPHURIC ACID.

By M. E. BARRAL.

WHEN the powdered parabichloride of hexachloridised benzene, $\text{C}_6\text{Cl}_6\text{Cl}_2^{1-4}$, is dissolved gradually in sulphuric acid containing disulphuric acid, a magnificent reddish violet colouration is produced.

By adding water or ordinary sulphuric acid, or even by leaving the mixture in contact with moist air, the colouration disappears as soon as all the disulphuric acid has been transformed into SO_4H_2 . Under the influence of water and SO_4H_2 , C_6Cl_8 gives both perchloridised benzene, perchlorised quinone, hydrochloric acid, and free chlorine:—



To show that this colouration is not due to the impurities of the Nordhausen acid, and that it is not produced in the presence of the anhydride, I prepared some sulphuric anhydride synthetically by passing $\text{SO}_2 + \text{O}$ over spongy platinum. By grinding together C_6Cl_8 and SO_3 , being careful to exclude all moisture, nothing is produced, while a beautiful reddish violet colouration appears as soon as the mixture is exposed to moist air.

Volumetric Estimation.—The disappearance of the colouration at the moment when all the disulphuric acid is transformed into SO_4H_2 , enables us to make use of $\text{C}_6\text{Cl}_6\text{Cl}_2^{1-4}$ as an indicator in the volumetric estimation of disulphuric acid.

A given volume of Nordhausen acid (25 c.c. for example) is poured into a flask, a little powdered C_6Cl_8 is added, and the mixture is shaken up until the colouration appears. We then run in, drop by drop, some carefully titrated sulphuric acid (acid at 66° , to which is added one-tenth its weight of water), until the disappearance of the reddish tint: a simple calculation enables us to estimate the quantity of disulphuric acid present in the Nordhausen acid.

This method of estimation, which enables us to act directly on the acid, gives excellent results in cases when the acid is colourless or only faintly coloured. Unfortunately it loses a good deal of its sensitiveness with commercial Nordhausen acids; these being as a rule more or less yellow, or even brown, the disappearance of the reddish tint is difficult to observe very accurately.—*Journ. de Pharm. et de Chim.*, Series 6, vol. vi., No. 3.

CONTRIBUTION TO THE STUDY OF THORIUM.

By G. URBAIN.

1. WHEN we wish to obtain thorium from thorite, we treat the latter with hydrochloric acid and evaporate to dryness to render the silica insoluble. We separate the lead and the tin by means of sulphuretted hydrogen, which is afterwards driven off by boiling. We then add a little chlorine-water to peroxidise the iron before precipitating it with oxalic acid, if the mineral does not contain the alkaline earths in any notable quantity. The oxalates are then calcined, and the oxides converted into

anhydrous sulphates, which are dissolved by projecting them—a little at a time—into iced water.

This is rather a delicate operation; above all, when working on large quantities, because the sulphates of the cerium group have a strong tendency to become hydrated, and for this reason are very difficult to dissolve. It is therefore necessary to take great care to rid these sulphates of all trace of free acid, and to add them to the water in minute quantity only if we wish to get them entirely dissolved. It has been noticed that a little acetate of ammonia increases the solubility of the sulphates of this group enormously.

A cold saturated solution of acetate of ammonia, diluted with twice its bulk of water, dissolves flocculent sulphate of thorium instantly. The double potassic sulphates of thorium, cerium, lanthanum, and didymium also dissolve with great ease in the same reagent, and they are not precipitated on boiling.

The sulphate of thorium containing $8\text{H}_2\text{O}$, when treated with a saturated solution of acetate of ammonia, forms a felty mass composed of fine needles of acetate of thorium. In a weaker solution it is completely dissolved. It is slightly precipitated on boiling, and at the same time acetic acid is given off, and the turbidity does not disappear by adding water or acetic acid, but only on the addition of hydrochloric acid. The presence of free acetic acid diminishes the solubility of the acetates very considerably.

Formate of ammonia appears to behave in a very different manner, and I intend to return to this subject later on.

The method of purifying thorium, as described by Nelson, is long and tedious. It consists of precipitating the thorium in the state of a flocculent sulphate, and it gives very bad results.

The solution of the oxalate in oxalate of ammonium carries with it a large proportion of cerium. The hypsulphite method gives results hardly better than the incomplete precipitations obtained by alkalis. None of these methods give even approximately pure thorium, except by repeating the operations several times.

2. I obtained thorium, which did not show a trace of cerium, very rapidly by the action of peroxide of hydrogen on its hydrate, with the formation of an acetylacetonate. For this purpose I used a relatively pure thorium which I had prepared in the following manner:—The raw oxalates were treated with oxalates of ammonia, and instead of precipitating the oxalate from this solution by means of an acid, I found it more advantageous to precipitate it by ammonia.

The acetylacetonate of thorium was obtained very easily by treating the hydrate of thorium, suspended in dilute alcohol, by acetylacetone, evaporating to dryness on the water-bath, and taking up with chloroform, which dissolves the acetylacetonate of thorium and leaves it, on slow evaporation, in well-defined crystals.

The molecular weight was taken in bromide of ethyl by the cryoscopic method.

Substance used	1.308 grms.
Weight of solvent	94.157 "
Loss of weight	0.26 "

	Found.	Calculated.
Molecular weight	630	628.4

This was done by taking the atomic weight found by Nelson, viz., 232.4.

I have also estimated the thorium by treating the acetylacetonate with nitric acid and then calcining the nitrate:—

Substance used	0.570 grm.
Thoria found	0.241 "

	Found.	Calculated.
Per cent.	42.2	42.1

Combustion gave me the following figures:—

Substance used	0.570 grm.
Water	0.115 "
Carbonic acid	0.389 "

Summary of Analyses.

Found.		Calculated.		
Th.. .. .	37.09	36.98	79.62	
C	37.75	38.19		
H	4.54	4.45		
O	4.54	20.38		
Total.. .. .	100.00			

The acetylacetonate of thorium therefore corresponds to the formula $\text{Th}(\text{C}_5\text{H}_7\text{O}_4)_4$.

Under similar conditions, the hydrate of cerium is transformed into a grey amorphous body, almost insoluble in chloroform and alcohol. The analysis of this body shows it to be an ill-defined compound very like a basic salt—



We can obtain acetylacetonate of thorium just as easily by the double decomposition which takes place between acetylacetonate of soda and any salt of thorium. The liquid is well shaken up with chloroform, which is then distilled off.

3. Acetylacetonate of thorium, though but slightly soluble in water, is dissolved by most organic solvents. It is very soluble in alcohol and chloroform, but less so in ether and bromide of ethyl. Its crystals are of the clinorhombic system, and when formed from ether or chloroform they appear as elongated prisms:—

$$\begin{aligned} e' : e' &= 82^\circ 16' \\ e' : m &= 30^\circ 05' \\ m : m &= 80^\circ 55' \end{aligned}$$

When formed from bromide of ethyl the crystals are only microscopic.

Acetylacetonate of thorium melts at $171-172^\circ$ and sublimes *in vacuo*.—*Bull. Soc. Chim.*, Series 3, vol. xv., p. 347.

THE SEPARATION OF ALUMINUM AND BERYLLIUM BY THE ACTION OF HYDROCHLORIC ACID.*

By FRANKE S. HAVENS.

In a former paper (Gooch and Havens, *Am. Journ. Sci.*, ii., December, 1896) a method was described for the determination of aluminum in the presence of iron, based upon the fact that the hydrous aluminum chloride, $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$, is practically insoluble in a mixture of strong hydrochloric acid and anhydrous ether saturated with hydrochloric acid gas, while the ferric chloride is entirely soluble in that medium.

The work to be described in this paper is an extension of this process to cover the separation of aluminum from beryllium, with the subsequent determination of the beryllium by weighing as the oxide after conversion to the nitrate and ignition.

The aluminum chloride solution was prepared by dissolving the so-called pure aluminum chloride of commerce in as little water as possible, precipitating, and washing free from iron with strong hydrochloric acid, dissolving the chloride thus obtained in water, precipitating the hydroxide by ammonia, washing the precipitate free from all alkalis, and re-dissolving it in hot hydrochloric acid. From this solution, after cooling, gaseous

hydrochloric acid precipitated the pure hydrous chloride. This prepared chloride was dissolved in water and the solution standardised by precipitating with ammonia the hydroxide from weighed portions and weighing as the oxide. The solution of beryllium used was made by dissolving in water beryllium chloride found to be free from iron by the sulphocyanate test, and giving no precipitate when tested by the gaseous hydrochloric acid process, to be described later on. This was standardised by precipitating with ammonia the hydroxide from weighed portions and weighing the ignited oxide in the usual manner.

In the experiments of Table I., weighed portions of the aluminum solution were mixed with portions of the beryllium chloride solution representing from 0.01 to 0.10 grm. of the oxide, an equal volume of a mixture of strong hydrochloric acid and ether (taken in equal parts) was added to the solution of the mixed chlorides, and the whole was completely saturated with gaseous hydrochloric acid while kept at a temperature of about 15°C . by immersing the receptacle in running water. Ether was added, equal in volume to the aqueous aluminum and beryllium solutions originally taken, and the current of gas again turned on until saturation was complete. By this treatment there is present at the end of the saturation a volume of ether equal to that of the aqueous hydrochloric acid introduced and generated. The finely-crystalline precipitate of aluminum chloride was caught on asbestos in a filter crucible, washed with a previously-prepared mixture of hydrochloric acid and ether in equal parts, saturated at 15°C . with hydrochloric acid gas, and dried for half an hour at a temperature of 150°C . It was next covered with a layer of pure mercuric oxide, which had been tested and found to leave no residue on volatilising, and the crucible was gently heated over a low flame under a ventilating hood and finally ignited over the blast.

TABLE I.

	Al_2O_3 taken in solution as the chloride.	Al_2O_3 found.	Final volume. C.m.s.	Error.
1.	0.1046	0.1044	12	0.0002—
2.	0.1046	0.1038	12	0.0008—
3.	0.1067	0.1066	12	0.0001—
4.	0.1071	0.1063	12	0.0008—
5.	0.1059	0.1054	30	0.0005—

From these results it is obvious that the aluminum chloride may be determined in the presence of beryllium chloride with reasonable accuracy.

The beryllium may be recovered in the filtrate from the aluminum chloride by precipitation with ammonia after nearly complete evaporation of the acid. It was found, however, upon trial that the conversion of the chloride to the oxide without precipitation and filtration may be easily accomplished by treatment with nitric acid and ignition. The results of Table II. indicate this clearly. In these experiments weighed portions of the beryllium solution were evaporated just to dryness on a radiator, care being taken not to heat to the volatilising-point of the beryllium chloride, a few drops of strong nitric acid were added, the liquid was evaporated, and the residue heated—at first gently, to break up the nitrate safely, and finally on the blast. It was found that this conversion of the beryllium to the nitrate can be carried on in platinum without attacking that metal appreciably, providing care be taken to remove thoroughly all excess of hydrochloric acid before the nitric acid is added to the dry residue.

TABLE II.

	BeO taken in solution as the chloride.	BeO found.	Error.
1.	0.0483	0.0481	0.0002—
2.	0.0483	0.0483	0.0000
3.	0.1076	0.1085	0.0009+

In Table III. (1 to 9) are given the results of experiments in which both the aluminum and the beryllium

* Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, Series 4, Vol. iv., No. 20, August, 1897.

were determined—the former by precipitation as the hydrous chloride and weighing as the oxide after igniting with mercuric oxide; the latter by the conversion of the chloride, through the nitrate, into the oxide. In Expt. 10 (made to get a comparison of the methods) the beryllium was recovered by precipitating the hydroxide with ammonia from the partially evaporated solution of the chloride after removing the aluminum.

In Experiments 1 to 5 inclusive, the aluminum was determined exactly as previously described; in 6 and 7, the solutions (being originally larger) were concentrated by evaporation previous to the addition of the ether and hydrochloric acid mixture. In Experiments 8, 9, and 10 the treatment was varied advantageously by saturating the aqueous solution directly with hydrochloric acid gas before adding an equal volume of ether, and completing the saturation.

TABLE III.

	Al_2O_3 taken in solution as the chloride.	Al_2O_3 .	Error.	Final volume.	BeO taken in solution as the chloride.	BeO found.	Error.
				Cm. ³ .			
1.	0.1059	0.1058	0.0001	12	0.0198	0.0204	0.0006+
2.	0.1053	0.1044	0.0009	12	0.0194	0.0196	0.0002+
3.	0.1065	0.1059	0.0006	12	0.0197	0.0205	0.0008+
4.	0.1068	0.1060	0.0008	12	0.0199	0.0207	0.0008+
5.	0.1049	0.1047	0.0002	12	0.0198	0.0208	0.0010+
6.	0.1060	0.1057	0.0003	12	0.0977	0.0969	0.0008-
7.	0.1064	0.1063	0.0001	12	0.1085	0.1084	0.0001-
8.	0.1046	0.1038	0.0008	30	0.1083	0.1087	0.0004+
9.	0.1051	0.1048	0.0003	30	0.1071	0.1078	0.0007+
10.	0.1076	0.1075	0.0001	30	0.1086	0.1094	0.0008+

These results are plainly very good.

The manipulation of the process is not difficult. The gaseous hydrochloric acid is most conveniently produced by the well-known method of treating with strong sulphuric acid, in regulated current, a mixture of strong aqueous hydrochloric acid and common salt. A platinum dish hung in an inverted bell-jar, provided with inlet and outlet tubes through which the current of water for cooling is passed, makes the best container for the solution to be saturated with the gas. It is advantageous to arrange the filtration upon asbestos, so that the filtrate and washings may be caught directly in the crucible (placed under the bell-jar of the filter-pump) in which the subsequent evaporation is to be effected. The heating of the strong acid solution must be gradual and conducted with care to prevent mechanical loss by a too violent evolution of the gaseous acid.

It only remains to thank Professor Gooch for kind suggestions and advice.

ON THE OCCURRENCE OF VANADIUM IN SCANDINAVIAN RUTILE.*

By B. HASSELBERG.

THE problem of assigning to each chemical element its definite emission spectrum has been carried perceptibly nearer to its solution, since the necessary basis was established by the classical work of Rowland. Yet when the attempt is made to extend it to the fainter radiations, as well as to the principal lines of the spectrum, the question becomes complicated to such an extent that an exhaustive solution seems to be well nigh hopeless. It is therefore necessary, in this department of spectroscopic

research, to be contented with a series of approximations; and no very great surprise need be felt if in a single case, where every effort has been made to eliminate such impurities as were most likely to occur, other impurities have been encountered, the presence of which there was in the beginning scarcely any reason to suspect. The present lines are devoted to a preliminary account of such a case as the above.

For producing the spectrum of titanium in the electric arc I have used titanic acid in the form of rutile with materially better success than when commercial titanium was employed, since the metal burns much too quickly when introduced into the arc, and is scattered in all directions. This rutile, which was kindly obtained for me by Baron Nordenskiöld, comes from Kragerø, in Norway. As in other kinds of rutile, its chief component is titanic acid, since, according to analysis of a number of varieties of this mineral (Dana's "Descriptive Mineralogy," 5th edition, 160, New York, 1883), the only other constituent to be expected is oxide of iron in the proportion of from 1 to 2 per cent. After eliminating the iron lines which are thus caused to appear on the spectrograms, as well as other metallic lines, whose presence was revealed by comparison with my own investigations of metallic spectra and with those of Kayser and Runge, I considered myself justified in ascribing the remaining lines to pure titanium, or at least in considering that only a few isolated cases remained in which contamination by a foreign metal might subsequently be proved. The continuation of my spectroscopic researches has shown, however, that this does not entirely hold good, since among the fainter and faintest lines of my titanium spectrum there are several that doubtless belong to vanadium. Having obtained through Baron Nordenskiöld a large piece of this metal, which was made by Moissan, of Paris, in the electric furnace, I recently began a re-examination of the spectrum, and discovered several strong groups of lines in the blue and violet parts, the approximate wave-lengths of which agreed very closely with those of faint lines previously measured in the spectrum of titanium. In order to obtain a final decision on this point, the parts of the spectrum in question of vanadium and rutile were photographed in juxtaposition on the same plate, in the usual manner, and compared line by line. The result of this investigation of approximate coincidences is shown in the accompanying table.

In the first two columns of this table are given the approximate wave-lengths and intensities of vanadium lines, in the region $\lambda 403$ — $\lambda 405$, for which corresponding fine lines occur on the comparison plates of the spectrum of Norwegian rutile. These lines are given, with their intensities, in the two following columns, the wave-lengths being those of my former catalogue of titanium lines. As will be seen, the coincidences are almost all exact; and hence the corresponding faint titanium lines are to be removed from the titanium spectrum as properly belonging to vanadium.

I must confess that I have been greatly surprised by this result. It will be granted, I think, that there were no possible reasons for suspecting it in advance, since vanadium has never been found in any of the numerous varieties of rutile hitherto known. Under these circumstances it seemed to me desirable to subject another kind of rutile to the same test. For this purpose Swedish rutile from Kåringbräcka in Westmanland was chosen, one reason among others for doing so being that the results of Ekeberg (*Svenska vetensk. Akad. Handl.*, xlvii, 1893; Dana's "Mineralogy"), according to which this particular kind also contains chromium, could be tested at the same time. A double exposure to the spectrum of this mineral and of vanadium in the region $\lambda 425$ — $\lambda 445$ having been made, the same series of coincidences was found as in the case of the Norwegian rutile (see the fifth and sixth columns of the table), and hence at the same time the fact was demonstrated that Swedish rutile also contains vanadium.

* "Über das Vorkommen des Vanads in den Skandinavischen Rutilarten," *Bihang till Svenska vetensk. Akad. Handl.*, xxii., Afd. 1, No. 1. From the *Astrophysical Journal*, v., No. 3.

Vanadium.		Rutile I.		Rutile II.		Remarks.
λ	i.	λ	i.	λ	i.	
4033.03	1.2	..	..	..	..	Also a weak line in Ti.
90.70	3	90.73	1	..	..	Coincident, belong to Va. All the rutile lines are weaker on the comparison plate than here represented.
92.87	3	92.83	1.2	..	..	
95.60	3	95.65	1	..	..	
4100.00	3.4	4099.94	1.2	..	..	
05.30	3	05.31	1.2	..	..	
09.95	3.4	09.92	1.2	..	..	
12.00	4	11.91	2.3	..	..	
15.30	3.4	15.32	2	..	..	
16.65	3	16.64	1.2	..	..	
23.65	3	23.68	2.3	..	..	
28.25	3.4	28.20	2	..	..	Divided. λ Ti > λ Va.
31.35	1	31.38	1.2	..	..	Coinc. Belongs to Va.
34.60	3.4	34.60	1.2	..	..	Divided. λ Ti < λ Va.
59.87	2.3	59.79	2.3	..	..	Coinc. Belongs to Va.
69.45	1.2	69.46	2	..	..	Divided. λ Ti < λ Va.
83.45	..	83.45	1.2	..	..	Clearly divided. λ Ti > λ Va.
4227.95	2	27.80	2	..	..	Probably divided. λ Ti > λ Va.
68.85	3	..	1	..	1	Widely separated.
71.80	3	..	1	..	1	All these lines occur on the rutile photographs with the observed intensities. The blanks in the columns 3 and 5 of wave-lengths indicate that these lines do not occur in my catalogue of the titanium lines. On the plates here investigated they occur with the given intensities.
4330.15	3	..	1-	..	1+	
33.00	3	..	1-	..	1+	
41.15	3	..	1+	..	1.2	
53.05	3.4	53.01	1	53.01	1.2	
79.45	4.5	79.40	2	79.40	3	
84.90	4.5	84.85	2	84.85	2.3	
90.15	4.5	90.11	2	90.15	2+	
95.40	4	..	2	..	2	
4400.75	4	00.74	1.2	00.74	2	
06.85	4.5	..	1.2	..	2	Clearly divided. λ Ti > λ Va. Perhaps. λ Ti > λ Va. Probably divided. λ Ti > λ Va.
07.90	4.5	07.85	1.2	07.85	2+	
08.40	4	08.39	1.2	08.39	2+	
08.65	4.5	08.70	1.2	08.70	2+	
16.65	3	16.70	2	16.70	2	
41.90	3.4	41.86	1.2	41.86	..	
44.40	3.4	44.41	3	44.41	..	

If, further, the observed intensities of the vanadium lines which occur in the photographic spectra of the two kinds of rutile are compared, it will be seen that the intensities for Rutile II., obtained at K ringbricka, are greater throughout the spectrum than those for Rutile I. Since this fact appears to indicate that Swedish rutile contains a greater amount of vanadium than Norwegian, it was of interest to test the relation of the two varieties in this respect by a special experiment, in which the exposure and development were exactly the same. With this object, two exposures in the upper region of the spectrum were made on the same plate, using for each a different half of the slit, with electrodes which in one case were made of Norwegian and in the other case of Swedish rutile. The exposure was in each case 1.5 minutes. The developed plate showed the titanium lines with identically the same intensity in both spectra, while the vanadium lines were considerably stronger in the spectrum of the Swedish rutile. In order that this fact may be clearly brought out, I have made a photographic copy of a drawing, in which the appearance of the negative under the microscope of the measuring engine is represented with all possible exactness. By this it will be seen that vanadium lines of the Norwegian rutile have a distinctly lower intensity, so that, in fact, some of the weakest of them fail to appear.

From what has been given above, I believe that it may be regarded as proved that both kinds of rutile contain vanadium, the Norwegian as well as the Swedish, but that the Swedish variety contains a considerably greater amount of this metal than the other. Whether this amount of vanadium is great enough to be recognised or quantitatively determined by ordinary chemical analysis is a question for the solution of which the above experiments afford no data, or at least only such as are highly

uncertain, for we have as yet no trustworthy information as to the sensitiveness of the spectral reactions of the elements.

On the plates which contain the spectra of the two kinds of rutile, the correspondence of lines (leaving out of consideration the difference of intensity of the vanadium lines already mentioned) is complete, with one exception. This exception is found in three quite strong lines which occur in the spectrum of the Swedish rutile, but of which there is scarcely a trace in the other variety. By referring their positions to neighbouring titanium lines I obtained for these lines the following wave-lengths:—

$$\lambda = 4254.50 \\ 74.90 \\ 89.90$$

while the strongest lines in the whole chromium spectrum have, according to my earlier measures, the wave-lengths

$$\lambda = 4254.49 \\ 74.91 \\ 89.87$$

The lines therefore belong to chromium, the presence of which is thereby demonstrated, and this result is in agreement with the analysis of Ekeberg.

Probable Antiquity of Mining for Tin in Bretagne.
—L. Davy.—All authors who have studied the ancient working of tin in the west of Europe admit that it was far anterior to the occupation of the country by the Romans, and think that the mines of Abbaretz-Nozay were abandoned by the Gauls about the date of the Roman invasion.—*Comptes Rendus*, cxxv., No. 5.

A CRITICAL REVIEW OF THE METHODS
OF DETERMINING MINERALS.*By Dr. JOSEPH W. RICHARDS,
Of the Department of Metallurgy and Mineralogy of the Lehigh
University.

THE determining of a mineral consists in finding out its species, and in doing this so conclusively as to prove that it is the species named to the exclusion of all other similar related minerals.

The question must be met at the outset, "What constitutes a mineral species?" I will answer by quoting our one-time professor of chemistry at the Institute, Dr. Persifor Frazer, whose word in geology and mineralogy carries with it the weight of an authority. Says Dr. Frazer, "Every true mineral is a definite chemical compound or element, homogeneous throughout its parts, and capable of expression in a formula. Its molecule is a distinctive whole—the unit of its mass—and incapable of division as long as the mineral retains its characteristic properties."

It is one of the easiest things in the world to accept the present scientific definition, and to forget the laborious steps by which such an apparently simple conclusion was evolved. Dr. Frazer's definition appears to us so nearly self-evident that it is difficult to realise that eighty years ago mineralogists were hotly debating the question as to whether the identity of a mineral consisted in its chemical composition or in the sum total of its physical properties. Thence arose two schools of mineral classification. Werner and his followers adopted the natural-history method of classification on the basis of physical properties alone, and prided themselves on having achieved a classification entirely independent of any aid from chemistry. The other school, of which Berzelius was one of the founders, insisted on the chemical composition as the basis of a proper classification, but were not so exclusive as the other school, and admitted physical distinctions, particularly that of form (*i.e.*, crystallisation), into their smaller subdivisions.

It will help, in our further consideration of the subject, to examine more closely these two schemes, particularly the Wernerian.

Cronstedt, about 1750, divided the mineral kingdom into four great divisions, viz.:—

- | | |
|----------------------|--------------------|
| I. Earth and stones. | III. Combustibles. |
| II. Salts. | IV. Ores. |

Werner, the first of the illustrious teachers of mineralogy at Freiberg, adopted this division and extended it greatly. He divided the first order into nine genera, as follows:—

- | | |
|----------------|----------------|
| <i>Earths.</i> | |
| 1. Zirconian. | 5. Calcareous. |
| 2. Siliceous. | 6. Barytic. |
| 3. Aluminous. | 7. Strontian. |
| 4. Magnesian. | |
| <i>Stones.</i> | |
| 8. Diamond. | 9. Hallites. |

Here, indeed, it would appear that the classification was nothing less than purely chemical, yet it was not. A certain number of external characters which siliceous minerals, for instance, usually exhibit being assumed as generic characters, or as a type of the genus, every mineral possessing those characters, whether it contains any siliceous or not, is arranged under the siliceous genus. And so with the other of the nine genera. Under these principles, Werner saw no inconsistency in placing sapphire in the siliceous genus and opal in the aluminous. I need not expatiate on the absurdity of this procedure from our modern standpoint, but it is only the stupendous development of chemical investigation in recent years that has lifted mineralogy out of that confusion.

Salts, according to Werner, included only such minerals as have some taste and a considerable degree of solubility in water; and they were divided into carbonates, nitrates, muriates, and sulphates.

Combustibles were divided into sulphur, bitumen, graphite, and resin.

Ores were divided into as many genera as there were distinct metals found as ores. Thus, all the iron ores constituted one genus. The genera were divided into species, depending on their external characters. This was a true chemical basis to start with, but far inferior to the classification according to the acid ingredient instead of the base.

The key-note of this system is found in the following quotation from Werner himself:—"Whenever the external characters and the chemical composition are at variance, the species is determined solely by the external characters." According to this method of reasoning, gypsum and selenite were classed as different species because they looked different, although of exactly the same composition, while calamine and smithsonite were classed together as one mineral because they looked alike, although of very different composition.

Coming to the chemical systems of classification, they were far from being above reproach, yet they showed fewer inconsistencies than the other method. Many mistakes were made, it is true, principally owing to the imperfect state of chemical science. The phenomena of dimorphism were yet awaiting the light of Mitscherlich's genius for their explanation, and the reproach was cast on the chemical method that it had to classify together calcite and aragonite as one species, while everybody knew they were different. Many were the hours spent by zealous adherents of the chemical side, in trying to establish by analysis some essential difference between calcite and aragonite, and thus to silence their opponents. Mitscherlich's researches on isomorphism and dimorphism, however, cleared up so many of these difficult points that in 1841 Rammelsberg was able to resuscitate the almost defeated chemical method, and to champion it so effectively as to have made it the present accepted basis of mineral classification.

A very striking example of this change of base in mineral classification is shown in the successive standard American works on mineralogy. Shepard's work, issued in 1835, 1844, and 1857, in successive editions, was on the natural-history method. J. D. Dana's first edition, in 1837, followed the same system; the second edition, in 1844, was similar, but described in an appendix Rammelsberg's chemical system; the third edition, in 1850, discarded the natural-history method of classification, and adopted altogether the chemical method. This has been retained in all subsequent editions of this magnificent work, which has become not only the American authority on mineralogy, but, one may almost say, the international authority. The importance of this classification to our discussion merits that it be given at once, in outline, as follows:—

Dana's Classification of Minerals.

- I. Native elements.
- II. Sulphides, arsenides, antimonides, selenides, and tellurides.
- III. Chlorides, bromides, and iodides.
- IV. Fluorides.
- V. Oxygen compounds.
 - (1) Oxides.
 - (2) Ternary oxygen compounds, or oxygen salts.
 1. Silicates.
 2. Tantalates and columbates, &c.
 3. Phosphates, arsenates, vanadates, &c.
 4. Borates.
 5. Tungstates, molybdates, chromates, &c.
 6. Sulphates.
 7. Carbonates.
- VI. Hydrocarbons.

* The Journal of the Franklin Institute, cxliv., p. 139.

It is now proper to inquire into the subject of the identification of a mineral, *i. e.*, its determination.

The question is how to most quickly, easily, and surely identify an unknown specimen. This is not exactly the same sort of question as the one of classifying properly a mineral after it is completely described; they are two aspects of the same problem, but from different points of view; for, as Dr. Sterry Hunt has said, "A natural system of classification is not subordinate to the end of identifying species, but should consider objects in all their alliances and relations." Systems for identifying species are not therefore necessarily along the identical lines of mineral classification. However, the natural-history method of classification by outward characters adapts itself very readily to schemes of identification in which the physical properties are given the pre-eminence in determining the mineral. Determinations of hardness, streak, lustre, gravity, fusibility, form, &c., are easily made without any special training, and therefore are most useful to beginners in the science of determining minerals. The chemical system of classification, however, at once suggests chemical analysis as the true basis of a determinative method, and lends itself most kindly to that means of identification. Moreover, the methods of chemical analysis, particularly those of blowpipe analysis, have developed coincidentally with the growth of the chemical classification of minerals.

The postulate may be made, that if every mineral species is a definite chemical compound, then the true basis of identifying the mineral should be the chemical analysis. In many cases this may not be the quickest way, but it is bound to be the surest. But shall we throw away all the aid to be derived from physical tests? By no means. If from the properties at once evident on casual inspection memory suggests a familiar mineral, let chemical analysis at once come in to confirm or deny the determination. In the great majority of cases the physical determination is practically valueless without the analytical confirmation; and there are very few cases in which the chemical confirmation is absolutely unnecessary or useless. On the other hand, whenever a mineral is not at once recognised on inspection, the true basis of search to determine it should be to fix as nearly as possible its composition by chemical analysis, and then to utilise physical tests to distinguish between minerals difficult or impossible to separate by differences in their composition.

Tables based entirely on physical tests have been constructed; mixed tables, based on a combination of both physical and chemical tests, have been devised; I have preferred to attack unknown minerals *entirely* from the chemical side, and will later on explain what appear to me the advantages of this method.

It is, of course, superfluous to analyse in detail every one of the numerous schemes of determinative mineralogy which have been constructed; it will suffice to select a few typical examples, and to discuss them on general principles.

Dana gives in an appendix to his text-book on Mineralogy one set of tables based primarily on the crystal system, then on lustre (metallic and non-metallic), and then with the minerals arranged according to specific gravity. This classification is very useful if the mineral shows clearly its crystallisation, and is truly of the system which it appears to be; but every mineralogist knows that minerals are oftener uncrystallised than crystallised, and that in probably one case out of three the system is not what it appears to be. The question of lustre may sometimes mislead. The specific gravity may be depended upon in probably three cases out of four. Summing up the probabilities that a mineral could be identified by the use of such a table, they are about one in two if the specimen is crystallised, and not more than one in ten if it is uncrystallised. In scarcely any case would the identification be completely satisfactory without the chemical confirmation.

A set of tables in more extensive use than Dana's, and

based also on physical tests alone, is that of Dr. Weisbach, well known in English by Dr. Frazer's translation. The following outline shows the scheme of this classification:—

Weisbach's Tables.

- I. Minerals with metallic lustre.
 1. Red. 2. Yellow. 3. White. 4. Grey. 5. Black.
- II. Minerals of sub-metallic or ordinary lustre, and with coloured streaks, as follows:—
 1. Black. 2. Brown. 3. Red. 4. Yellow. 5. Green. 6. Blue.
- III. Minerals of ordinary lustre, with white or grey streak.
 1. Very soft. 2. Soft. 3. Semi-hard. 4. Hard. 5. Very hard.

Supplementary table for Class III.:—

- A. Soluble in water.
- B. Effervescing in HCl (carbonates).
- C. Not soluble in water or containing CO₂.
 1. Containing water—Easily fusible to infusible.
 2. Anhydrous—Easily fusible to infusible.

In using these tables, if there is no question as to the lustre, or colour, or streak or hardness, that is, if all these properties are normal and observed correctly, the specimen thereupon falls into a class varying in number from five to sixty-three, among which it is to be distinguished by a critical comparison of its other physical properties. In the case of the class of five, this is an easy matter; in the majority of cases, the classes averaging thirty to fifty, this is an arduous or impossible task. We have further to consider that sometimes the lustre is doubtfully metallic, frequently the colour is not easy to judge, that the streak is often misleading, and, most of all, the hardness very seldom to be relied upon. I should say in all fairness, taking all these points into consideration, that a careful observer would not place more than fifty out of one hundred promiscuous specimens in the class to which Weisbach assigns them, and, out of those fifty correctly classed, would not be able to identify over twenty-five with any reasonable degree of certainty without having recourse to the chemical composition for confirmation.

One of the best examples of tables based on mixed physical and chemical tests is that of Von Kobell, which is the basis of the classification so well known and largely used in the United States, *viz.*, Brush's. They are as follows:—

Von Kobell's Tables.

- I. Metallic lustre.
 - a. Native malleable metals and mercury.
 - A. Fusible at 1 to 5 or easily volatile, containing—
 1. Arsenic. 2. Selenium. 3. Tellurium. 4. Antimony. 5. Sulphur. 6. None of these.
 - B. Fusible above 5, or infusible, not volatile.
 1. Containing manganese. 2. Magnetic B.B. 3. All others.
 - II. Without metallic lustre.
 - A. Easily volatile or combustible.
 - B. Fusible at 1 to 5.
 - (I.) Reduces to metallic button, or magnetic B.B.
 1. Silver. 2. Lead. 3. Copper {with As. no As.
 4. Cobalt. 5. Magnetic B.B. 6. All others.
 - (II.) Not belonging to (I.)
 1. Alkaline after ignition.
 2. Soluble in HCl, without residue.
 3. Soluble in HCl, with gelatinous residue.
 4. Soluble in HCl, with sandy residue.
 5. Insoluble in HCl, has Mn.
 6. All others.

- I. Metallic lustre.
- II. Without metallic lustre.
 - A. Easily volatile or combustible.
 - B. Fusible at 1 to 5.
 - C. Fusible above 5, or infusible.

1. With cobalt nitrate show alumina.
2. With cobalt nitrate show zinc.
3. React alkaline after ignition.
4. Soluble in HCl, contain no silica.
5. Soluble in HCl, contain silica.
 - (1). Hydrrous.
 - (2). Anhydrous.
6. Not belonging to the foregoing divisions.
 - (1). Hardness under 7.
 - (2). Hardness 7 or over.

It will be seen that the basis of this classification is primarily and secondarily physical, afterwards chemical composition plays an important part. The superiority of this method over the previous ones based solely on physical tests, *lies almost altogether in the extent to which the chemical tests are introduced.* There is still some uncertainty as to lustre being correctly determined, and considerable uncertainty is introduced by the factor of fusibility where it lies anywhere near to five; but, granting that these are correctly determined, then the chemical tests are reached and the uncertainties mostly cease. No one worthy of being called a chemist can mistake sulphur for selenium or silver for lead; the identification of such characteristic elements is practically a certainty. With such chemical tests as solubility in hydrochloric acid, with a gelatinous or sandy residue or no residue at all, there is still considerable room for uncertainty, and the scheme is weak in proportion as they are introduced.

Dr. Fuchs introduced mixed tables based primarily on chemical composition, and to that extent superior to Weisbach's. As a whole I have found these tables (or subsequent ones based upon them) to be the most satisfactory tables published. To observers skilled in blowpipe analysis they furnish the most satisfactory means of identification of an unknown specimen. The scheme is as follows:—

Fuchs's Tables.

(By the Blowpipe).

- I. Heated B.B. on charcoal.
 1. Volatiles or burns.
 2. Arsenic fumes { metallic.
non-metallic.
 3. Selenium fumes. 4. Shows antimony.
 5. Shows tellurium.
 6. Reacts alkaline { soluble.
non-soluble.
 7. Residue magnetic.
- II. Reduced B.B. with soda, on charcoal.
 1. Reacts for sulphur and gives metallic button.
 2. Reacts for sulphur and gives no metallic button.
 3. No sulphur reaction, but gives metallic button.
- III. Shows manganese in the beads { metallic lustre.
non-metallic lustre.
- IV. Shows zinc with cobalt solution.
- V. Soluble in HCl, without residue.
 1. Fusible B.B. (a). Hydrrous. (b). Anhydrous.
 2. Infusible B.B. (a). Hydrrous. (b). Anhydrous.
- VI. Soluble in HCl to a jelly (silica).
(Classified similarly to V.).
- VII. Soluble in HCl, with separation of silica (no jelly).
 1. Hydrrous. 2. Anhydrous.
- VIII. Insoluble in HCl, but bead test shows silica.
 1. Fusible B.B. 2. Infusible B.B.
- IX. Belonging to none of the previous divisions.

The weakest point of these tables is in the action of hydrochloric acid, where there is room for some uncertainty, and where tests for the elements, as in the previous divisions, are generally much more satisfactory. Division IV. should also be abolished, and placed as a fourth subdivision of II., for by reducing with soda and a little borax on charcoal, zinc is more certainly identified than by the cobalt nitrate test. Cadmium minerals could also have been included under the same subdivision. Taking these tables altogether, I would call them the most satisfactory of the published tables.

(To be continued).

NOTICES OF BOOKS.

Incompatibilities in Prescriptions; for Students in Pharmacy and Medicine, and Practising Pharmacists and Physicians. By EUSEB A. RUDDIMAN, Ph.M., M.D. New York: John Wiley and Sons. London: Chapman and Hall, Lim. 1897. Pp. vi.—267. 8vo.

THE author of this valuable work holds the Professorship of Pharmacy and Materia Medica in Vanderbilt University, Nashville, Tennessee, and, having the Degrees of Master in Pharmacy and Doctor of Medicine, is well qualified for his undertaking. There are two distinct divisions of the book. Part I. contains, in a condensed form, the more common incompatibilities, the substances being arranged in alphabetical order of their Latin names. Part II. contains 325 prescriptions, with a criticism of each explaining the difficulties of compounding, or the objections to the form in which they are written. In the first part the ordinary reactions of the given substance are stated, and the decompositions liable to ensue when brought into contact with other substances by trituration, and aqueous or alcoholic solution.

The several reactions in each paragraph are numbered for convenience of reference; thus *antipyrinum* is treated in twenty-three short items, and *acidum tannicum* in twenty-six items.

Carelessness on the part of the medical man, or on the part of the apothecary's assistant, may cause explosions when certain substances are triturated in a mortar or brought together in solution; the active substances may be entirely changed by oxidation, reduction, or double decomposition; an insufficiency of a solvent may interfere with the desired form of prescription; the order in which ingredients are mixed may greatly affect the result; the proportion of the substances may have to be altered to obtain the end sought; and some ignorant practitioners may actually send to the pharmacist prescriptions which cannot be filled without danger to the compounder or to the patient. All these cases are abundantly illustrated and carefully explained in the second part of the book, by citing actual prescriptions and criticising them. Prescription No. 36 calls for potassium chlorate, precipitated sulphur, sulphide of antimony, and sugar, and the author remarks that each substance should be powdered separately and mixed lightly, which is certainly a wise caution. Prescription No. 132 contains amyl nitrite, potassium iodide, alcohol, and syrup of lemons, with the direction *Cito dispensetur!* Dr. Ruddiman recommends that this should not be dispensed as written, but that the physician should be communicated with. Prescription No. 87 is said to be part of the Keely cure for alcoholism; it contains chloride of gold and sodium, sulphate of strychnine, sulphate of atropine, and fluid extract of cinchona.

The author suggests that the student of pharmacy study each prescription thoroughly before consulting the note accompanying it.

The work shows care throughout, but the author overlooks the formation of sodium nitrate in his note to prescription No. 170. Two indices complete the well-printed volume, one to incompatibilities and one to prescriptions.

It would be interesting to learn the sources of the prescriptions cited by the author; they are presumably from the note-books of apothecaries and from published medical treatises, a list of which he gives as the authorities consulted. To the lay mind many of them appear to be horrible mixtures, almost as objectionable as the nostrums of Paracelsus and his followers; and the question arises, Does the physician who prescribes substances that mutually decompose when mixed (sometimes generating bodies quite different from their original forms) take into consideration the physiological action of the newly-formed compounds, or does he attribute the effects to the primary ingredients?

This volume will be invaluable to students of pharmacy and to young practitioners; but is not its existence a commentary on the deficient education in the fundamental truths of chemistry of those licensed to compound and to administer medicines to a long-suffering people?

H. C. B.

Sessions of the Superior Board of Health, corresponding to the Year 1896. ("Sesiones del Consejo Superior de Higiene Publica, correspondientes al Año de 1896"). Santiago de Chile.

CONSIDERABLE attention seems to have been paid to the potable waters of Valparaiso. There have been in that city many cases of typhoid fever and of acute intestinal catarrh; the public water-supply passes through seats of infection. It was pronounced necessary that Santiago should be severed; that it is not possible to use for the disinfection of the sewage-waters the chemical procedures already in use, and that it is impossible to disinfect all the irrigation-waters of Santiago.

A Detailed Course of Qualitative Chemical Analysis of Inorganic Substances, with Explanatory Notes. By ARTHUR A. NOYES, Ph.D., Assistant Professor of Chemistry at the Massachusetts Institute of Technology. Third Edition. London and New York: Macmillan and Co., Lim. 1897. 8vo., pp. 89.

We have here what may be regarded as an abridgment of the well-known treatise on Qualitative Analysis by the late Prof. Remigius Fresenius. We can find little, if anything, to which we have any right to object. It seems to us slightly strange to find the author recommending that the laboratory work should be accompanied by recitations in which the process of analysis is discussed in detail.

CORRESPONDENCE.

MODERN ALCHEMY.

To the Editor of the Chemical News.

SIR,—As you have allowed Dr. Bolton to indulge in a little modern scientific witch-finding at my expense, I hope you will permit me to make the following rejoinder:—

1. My accuser says that I have "with boldness, publicity, and persistency," made claim to "success in transmutation or creation of gold." This charge is not in accordance with the facts of the case. Dr. Bolton is sufficiently well versed in modern newspaper methods to know that publicity frequently takes place in opposition to the desires of the parties concerned. He knows that this has largely been so with regard to my work respecting the interchangeability of the so-called "elements." He knows that the average reporter makes a surprising hash of scientific statements, and that the average catchpenny newspaper editor takes reprehensible liberties even with signed communications and interviews. He knows that when I was applied to respecting the accuracy or otherwise of the report printed in the *New York Journal*, I declined to admit that it was more than "substantially correct,"—a fact which surely should have precluded such report being quoted in your columns as my "own words."

2. The following correspondence has passed between my accuser and myself, viz.:—

a. Bolton to Emmens, June 21st, 1897:—

"I am preparing for an English paper an account of your work of converting silver into *argentaurum*, and write to enquire whether you can send me any later publication of your own than your announcement made in August, 1896.

"I will be obliged for any circular or other publication you have issued in respect to Ar."

b. Emmens to Bolton, June 22nd, 1897:—

"Replying to your letter of yesterday, I have to say that it will give me much pleasure to send you additional information. The little pamphlet will be ready in a few days, and then I will mail you a copy. It will, I think, interest you, even if you be not a Unitarian in material philosophy."

c. Bolton to Emmens, July 11th, 1897:—

"Your polite note was duly received, and I shall be very glad to receive a copy of your forthcoming pamphlet as soon as issued."

d. Emmens to Bolton, July 13th, 1897:—

"Yours of the 11th has reached me, and I now have the pleasure of sending you by this mail a copy of the just-completed pamphlet, '*Arcana Naturæ*'; and inasmuch as reference is made therein to my '*Argentaurum Papers No. 1*,' I also enclose a copy of that book."

e. Bolton to Emmens, July 16th, 1897:—

"I acknowledge with thanks your letter of July 13th, and your publications received to-day. I had heard of your work on Gravitation, but had not seen it, and shall examine it with interest.

"The '*Arcana Naturæ*' gives me just the information I needed. Thanking you for your courtesy, I am, &c."

The pamphlet alluded to in these letters is not a publication for sale. I prepared it in order to save time and trouble in replying to the numerous enquiries that are addressed to me from all parts of the world. It contains an explanation as to various developments and modifications that have taken place in my gold-work since the original announcement in the *New York newspapers*. Yet, with this explanation staring him in the face, my accuser, under the guise of reporting "*Recent Progress of Alchemy in America*," has served up, for the information of your eminent circle of readers, a mere copy of a popular article in a sensational newspaper published on August 18th, 1896, and has suppressed the particulars with which I civilly furnished him at his own request.

3. The pamphlet contains a clear and explicit account of a process of producing from Mexican dollars a substance that will pass muster as gold. My accuser withholds this from your readers, and then goes on to charge me with "secret processes" and "vagueness of description."

4. The pamphlet contains more than one explicit confession of my ignorance of natural things. Yet my accuser charges me with "an assumption of esoteric knowledge expressed in pseudo-philosophic language."

5. My accuser charges me with "an imperative present need of gold." In one sense of the words it may be conceded that every scientific man, as well as every scientific institution, is in need of gold. But this is not the sense in which Dr. Bolton wrote. No reader can be so obtuse as not to understand the "fling"; it is a piece of personal abuse for which my accuser cannot adduce one jot or tittle of justification. I have not appealed to him or to the public for one cent. I have, on the contrary, made him and other correspondents a free gift of information which has cost me many thousands of dollars to acquire. I think your readers will be of opinion that the learned gentleman owes it to them and himself to tender me an apology.

6. I will conclude with the narration of what may perhaps be deemed an interesting historical fact. On March 16th, 1897, I sent to the United States Assay Office, in New York, a Mexican dollar to be tested for gold. The report was *nil*. Thinking that this might have been an accidental case of individual purity, I then sent four Mexican dollars to the same Assay Office and had them there cut into halves. One set of halves was officially assayed with the same *nil* result as before. The other set was treated

in the Argentaurum Laboratory, without the addition of gold in any form, and the result was a relatively considerable production of a metal which answered to all the usual tests of gold, and was subsequently purchased as gold by the U.S. Assay Office.

For my own mental satisfaction I then instituted enquiries at the Assay Office as to the precise meaning to be attached to a report of *nil*; and I was informed that, where small weights of bullion are concerned, the report means that the amount of gold is less than one part in ten thousand—a proportion which, of course, was vastly exceeded by the gold actually produced by my treatment of the four half-dollars. One of two alternative conclusions was therefore compulsory:—Either some of the silver or copper in the dollars had been changed into gold or its simulacrum by my treatment; or the gold already existed in the dollars and was separated by my treatment, though not by the treatment in vogue at the U.S. Assay Office. I make every chemist a present of the dilemma. For my own part my “assumption of esoteric knowledge” does not carry me so far as to make me believe myself a more skilful assayer of bullion than Mr. Andrew Mason and his assistants.—I am, &c.,

STEPHEN H. EMMENS.

Argentaurum Laboratory,
20, Central Avenue, New Brighton,
Staten Island, New York, U.S.A.,
August 16, 1897.

[A specimen of argentaurum sent me by Dr. Emmens has been examined in the spectrograph. It consists of gold with a fair proportion of silver and a little copper. No lines belonging to any other known element, and no unknown lines, were detected.—W. C.]

HYPONITROUS ACID.

To the Editor of the Chemical News.

SIR,—As you have published in the *CHEMICAL NEWS* a translation of Hautzsch and Kaufmann's paper upon Hyponitrous Acid, which appeared in *Liebig's Annalen*, you will, I am sure, permit me to point out, also in the *CHEMICAL NEWS*, some errors which have entered into that paper, through its authors' neglect to read the literature of the subject.

Hautzsch and Kaufmann say that Maumené discovered hyponitrous acid. The facts are, however, as I have fully shown in the *Annalen*, that I am the undisputed discoverer, and that Maumené never experimented on the subject at all, and actually denied the existence of the acid, on purely theoretical grounds (*CHEM. NEWS*, xxiii., 206; xxv., 153 and 285).

The method for preparing hyponitrous acid, which the authors call Zorn's, and claim to have perfected (with what propriety will be shown some other time), is my method, my original method,—that is, for I have, in conjunction with Haga, published two others.

Piloty's method, referred to by the authors, had been, seven years before, anticipated by Haga and me (*Trans. Chem. Soc.*, lv., 760) in a much simpler and less expensive form, with the same satisfactory yield of more than half the theoretical quantity. In place of benzo-sulphidroxamic acid, we had used sulphidroxamic acid (oxyamidosulphonic acid); that was all the difference. The equation in our case was—



and that in Piloty's case,—



Concerning ammonium hyponitrite the authors should properly have referred to me as authority rather than to Zorn, since I had stated so many years earlier (*CHEM.*

NEWS, xxiii., 208) that ammonium chloride and silver hyponitrite give silver chloride and an alkaline solution which at once evolves ammonia.

The induction of the reaction of a hyponitrite with ferrous sulphate as a nitrate or nitrite by contact with undiluted sulphuric acid, which is made a point of by the authors, had already been recorded by me.

There are two important erroneous experimental observations contained in the paper, which the authors would have saved themselves from making had they become sufficiently acquainted with the literature of their subject before publishing, but the consideration of these I reserve till I publish the sequel to my first paper.

I wish to state that the correctness of the contents of this letter is not disputed. Professor Hautzsch and also Mr. Piloty have each in turn courteously acknowledged their oversight, and Professor Hautzsch has, without my action, recognised one of the errors as to experimental fact above alluded to.

It is the re-appearance of Hautzsch and Kaufmann's paper in the *CHEMICAL NEWS* which has obliged me to publish this letter. Also, to prevent misconception, I will add that no work upon hyponitrous acid—and there has been much—surpasses, if it equals, in value that done by the excellent Russian chemist Zorn, who died very young, to the great loss of chemistry.—I am, &c.,

EDWARD DIVERS.

Imperial University of Tokyo,
July 4, 1897.

PS. The number of the *CHEMICAL NEWS* which contains the beginning of Hautzsch and Kaufmann's paper has also a letter from Mr. Thomas Christie in commendation of taka-diaxase, so successfully discovered and studied by Mr. Takaminé, F.C.S. I take this opportunity to express the pleasure I feel in the fact that Mr. Takaminé is the first Japanese pupil I ever had, and that he pursued chemistry under me for four years.—E. D.

THE ALLEGED NEW ELEMENT IN IRON.

To the Editor of the Chemical News.

SIR,—I was much interested in Mr. Boucher's article in the last issue of the *CHEMICAL NEWS* (vol. lxxvi., p. 99), describing a possible element in pig-iron, and as I have come across a constituent of steel during the last few months resembling in every respect the description of its properties as given by Mr. Boucher, it may be of some interest if I give my experience.

A few months ago I received a small sample of steel drillings which had originally come from the Continent, and in the course of an analysis, in which, beside the ordinary constituents, appreciable quantities of arsenic, copper, cobalt, and nickel were found, I separated a dark brown sulphide in the arsenic group which had all the properties as described by Mr. Boucher—the most remarkable of them being the beautiful blue colour on evaporating down with sulphuric or hydrochloric acid. I had a very limited amount of the steel, and was working on 5 grms. After repeated precipitation and purification of the sulphide to eliminate any arsenic and antimony, the precipitate weighed 0.017 gm.; showing a very considerable proportion of this element—a quantity much too great to neglect. I therefore wrote to my clients requesting them to send me a further sample, as the steel contained a rare element which I wished to investigate further; this they were unable to do, and so the matter has stood in abeyance. The experiments I performed resembled closely those detailed by Mr. Boucher, with the exception that I used nitro-hydrochloric acid to dissolve the steel, instead of sulphuric acid.

At first sight it appeared to resemble molybdenum, but this was contradicted by many of its reactions, and I shall

await Sir William Crookes's report with much interest.—I am, &c.,

FREDK. G. RUDDOCK.

Laboratory and Assay Office,
19, Stanley Street, Warrington,
August 31, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 5, August 2, 1897.

Outset of the Combination between Hydrogen and Oxygen.—M. Berthelot.

Analysis of Aluminium and its Alloys.—Henri Moissan.

Fixation and Nitrification of Nitrogen in Arable Soils.—P. P. Dehérain.

Poisoning by the Sweat of a Healthy Man.—S. Arloing.—This paper shows that sweat contains substances energetically poisonous, possessing analogies with the certain microbial toxins.

On the Symmetric Tetramethyldiamidodiphenyl-dianthranaltetramethyldiamide of the corresponding Oxanthranol.—A. Haller and A. Guyot.—The conditions in which we have operated, the composition of the substance which we have obtained, enable us to conclude that we are in the presence of the diamidodimethylamido-phenyloxanthranaldimethyl.

Atomic Weights of Nitrogen, Chlorine, and Silver.—A. Leduc.—The author admits the following atomic weights:—O=16 (base), N=14.005, H=1.0076, Cl=35.470, Ag=107.916. As for sulphur we deduce from the experiments of Stas the atomic weight 32.056, he adopts this number, although the experiments of Dumas lead to 31.86, and those of Erdmann and Marchand to 32.005.

Thermo-chemical Determinations relating to the Cupric Compounds.—Paul Sabatier.—A thermo-chemical paper not suited for useful abstraction.

On certain Bromo-ketones.—A. Collet.—The author describes the parabromopropionyltoluene, the parabromobutyryltoluene, the bromopropionylparaxylene, the bromopropionylphenyl, and the chloride of dibromophenylpropionyl.

Observations on the Conjunction of the Diazo-derivatives with the Phenols.—Ch. Gassman and Henry George.—Not suitable for useful abstraction.

Carubinose.—J. Effront.—Carubinose appears as a syrupy substance, not crystallisable, soluble in water and alcohol, and answering to the formula $C_6H_{12}O_6$. It ferments readily with beer yeasts. The rotatory power of carubinose, its melting-point, and the crystalline form of its combinations with phenylhydrazin distinguish these sugars from the other monosaccharides.

On an Organic Compound rich in Manganese extracted from the Woody Tissue.—G. Guérin.—This compound, as obtained from beech-wood, yielded per cent.—C, 52.762; H, 5.04; N, 4.60; S, 0.666; P, 1.297; Mn, 0.402.

Moniteur Scientifique,
Series 4, Vol. xi., Part 1.

Progress realised in our Knowledge of the Constitution of the Alkaloids of Quinine.—Ch. Gassmann.—Not suitable for abstraction.

On the Applications of Electrolysis to Organic Chemistry.—L. Gourwitsch.—Already inserted.

Part 2.

On the rôle played by Peroxides in the Phenomena of Slow Oxidation.—A. Bach.—Already inserted.

Buletinul Societatii de Stiinte din Bucuresti
(Bulletin of the Scientific Society of Bucharest),
No. 2, 1897.

The Secretary-General, in a short obituary notice, announces the lamented death, after a painful illness, of C. Gogu, Professor of Mathematics in the University of Bucharest, and President of this Society, and he bears tribute to the loss the Society has sustained in his death. A biographical sketch of his life work will be printed in the *Annales* of the Society.

Subterranean Water in the North-west region of Bucuresilor.—N. Cucu St.—The author has sunk a number of shafts in various parts of the district, and has found water in considerable quantity in several places. He fully describes all the works carried out and gives excellent illustrations of some of the "fountains."

On a very Sensitive Reaction for Nitric Acid.—E. Riegler.—Reprinted from the *Pharmaceutischen Centralhalle*, Nos. 13 and 14, 1897.

Notes on the Constitution and Classification of the Sulpho-arsenical, Sulpho-antimonial, and Sulphobismuthic Minerals.—V. C. Butureanu.—There is, as is well known, an important group of minerals in nature known generally as sulpharsenides, sulphantimonides, and sulphbismuthides. The author has examined all of these that he has been able to obtain, and classified them according to their chemical formula, expressed graphically.

Classification of the Crystalline Rocks of the Central Zone of the Roumanian Carpathians.—L. Mrazec.—The rocks of this district are divided into three groups. The first comprises the distinctly crystalline rocks of the granitic gneiss type; the second, those of the well-crystallised mica-schists; and the third, the slightly crystalline chloritic schists. The second group is the one the author deals principally with in this paper.

ERRATUM.—P. 75, col. 1, line 32 from top, for "0.00366" read "0.00365."

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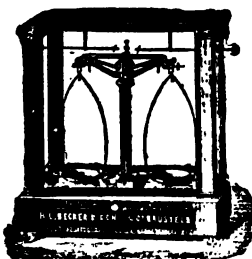
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JOURNAL OF PHYSICAL SCIENCE

Edited by [Sir Wm. Crookes, F.R.S.] (WITH WHICH IS INCORPORATED THE "CHEMICAL GAZETTE") [Established Fifty-three Years.]

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1972.

(STUDENTS' NUMBER).

A WORD TO STUDENTS.

IN spite of the recognised, and we might almost say the boasted, superiority of German chemists in the quantity of research yearly executed, and in their number of investigators, our Continental rivals have not the slightest disposition to rest and be thankful. In a pamphlet by Prof. Dr. Ferd. Fischer we find recommendations for further extending and securing their position in technical chemistry as compared with that of other countries. The author has here collected the views of his colleagues on the subject in question, and lays them before the world.

We find that there are in Germany four thousand technical chemists, exclusive of about two hundred others who study chemistry as a pure science. We cannot ascertain whether the figure of four thousand includes German technical chemists living and practising in other countries. We suspect that this is not the case. At most of the technical schools it is proposed to extend the curriculum, and to introduce examinations as a test for the proficiency of the students. Whether, and in how far, these proposals will lead to results of practical value time alone can decide. It will be noted that the course of study in the polytechnics and technical high schools varies greatly.

In many instances subjects are introduced which, however important in themselves, seem to us unnecessary in the special training of a technical chemist. We must keep in mind that, even for the most able and the most industrious student, there are only twenty-four hours in the day, and further that the great principle of division of labour must never be overlooked.

Instruction in law, with the single special exception of patent law, seems to us a needless encroachment on the spheres of the solicitor and the barrister.

We perceive that in some departments the candidate has a choice of subjects. Thus at Berlin, in the examination in chief, the student may select (a) geology, (b) spectrum analysis or general metallurgy.

We think that we in Britain are recovering some of the lost ground; but an immense field lies before us. Above all things we have to fight against the claims of the word-mongers; they are still far too strong and too influential to admit of our taking the foremost position in the world of Science.

UNIVERSITIES AND COLLEGES.

UNIVERSITY OF LONDON.

CANDIDATES for any Degree in this University must have passed the Matriculation Examination. No exemption from this rule is allowed on account of Degrees obtained or Examinations passed at any other University. This and all other Examinations of the University, together with the Prizes, Exhibitions, Scholarships, and Medals depending upon them, are open to Women upon exactly the same conditions as to Men.

There are two Examinations for Matriculation in each year; one commencing on the second Monday in January, and the other on the second Monday in June.

The Examination is conducted by means of Printed Papers; but the Examiners are not precluded from putting, for the purpose of ascertaining the competence of the candidates to pass, *viva voce* questions to any candidate in the subjects in which they are appointed to examine. These Examinations may be held not only at the University of London, but also, under special arrangement, in other parts of the United Kingdom, or in the Colonies.

Every candidate for the Matriculation Examination must, not less than five weeks before the commencement of the Examination, apply to the Registrar for a Form of Entry, which must be returned not less than four weeks before the commencement of the Examination, accompanied by a Certificate showing that the candidate has completed his sixteenth year, and by his Fee for the Examination. As no candidate can be admitted after the List is closed, any candidate who may not have received a Form of Entry within a week after applying for it must communicate immediately with the Registrar, stating the exact date of his application and the place where it was posted.

Every candidate entering for the Matriculation Examination for the first time must pay a Fee of £2 to the Registrar. If a candidate withdraws his name, or fails to present himself at the Examination, or fails to pass it, the Fee shall not be returned to him, but he shall be allowed to enter for any subsequent Matriculation Examination upon payment, at every such entry, of an additional Fee of £1, provided that he comply with the Regulations in the preceding paragraph.

Candidates are not approved by the Examiners unless they have shown a competent knowledge in each of the following subjects:—Latin. Any one of the following Languages:—Greek, French, German, Sanskrit, or Arabic. The English Language, and English History, with the Geography relating thereto. Mathematics. Mechanics. One of the following branches of Science:—Chemistry, Heat and Light, Magnetism and Electricity, Botany.

The Examination in Chemistry is—Chemistry of the Non-metallic Elements; including their compounds, their chief physical and chemical characters, their preparation, and their characteristic tests.

A Pass Certificate, signed by the Registrar, will be delivered to each successful candidate after the Report of the Examiners has been approved by the Senate.

If in the opinion of the Examiners any candidates in the Honours Division of not more than twenty years of age at the commencement of the Examination possess sufficient merit, the first six among such candidates will receive an Exhibition of thirty pounds per annum for the next two years; the second among such candidates will receive an Exhibition of twenty pounds per annum for the next two years; and the third will receive an Exhibition of fifteen pounds per annum for the next two years; such exhibitions are payable in quarterly instalments provided that on receiving each instalment the Exhibitioner declares his intention of presenting himself either at the two Examinations for B.A., or at the two Examinations for B.Sc., or at the Intermediate Examination in Laws, or at the Preliminary Scientific M.B. Examination, and Intermediate Examination in Medicine, within three academical years from the time of his passing the Matriculation Examination.

Under the same circumstances, the fourth among such Candidates will receive a prize to the value of ten pounds in books, philosophical instruments, or money; and the fifth and sixth will each receive a prize to the value of five pounds in books, philosophical instruments, or money.

Any candidate who may obtain a place in the Honours Division at the Matriculation Examination in January is admissible to the Intermediate Examination either in Arts or in Science in the following July.

INTERMEDIATE EXAMINATION IN SCIENCE.

The Intermediate Examination in Science will commence on the third Monday in July.

No candidate (with the exception of such as have obtained Honours at the Matriculation Examination in the preceding January) is admitted to this Examination within one academical year of the time of his passing the Matriculation Examination.

The Fee for this Examination is £5.

Examination for Honours.

Candidates for Honours in Chemistry will be examined in Inorganic Chemistry, treated more fully than in the Pass Examination. In addition, they will be examined practically in Simple Qualitative Analysis. This Examination will consist of six hours' examination by two printed papers and of six hours' practical work.

In the Examination for Honours, the Candidate, not being more than 22 years of age at the commencement of the Pass Examination, who most distinguishes himself will receive an Exhibition of £40 per annum for the next two years.

B.Sc. EXAMINATION.

The B.Sc. Examination will be held on the third Monday in October.

Candidates for this Examination are required to have passed the Intermediate Examination in Science at least one academical year previously.

The Fee for this Examination is £5.

Examination for Honours.

The examination for Honours in Chemistry will take place on Monday, Tuesday, and Wednesday in the week following the Examination for Honours in Mathematics; on Monday by printed papers (chiefly on Organic Chemistry), and on Tuesday and Wednesday by practical examination in Qualitative and Quantitative Analysis.

The candidate, being not more than 23 years of age, who most distinguishes himself in Chemistry, will receive £50 per annum for the next two years, with the style of University Scholar.

DOCTOR OF SCIENCE.

The examination for the Degree of Doctor of Science takes place annually within the first twenty-one days of June.

No candidate is admitted to the examination for the Degree of D.Sc. until after the expiration of two Academical Years from the time of his obtaining the Degree of B.Sc. in this University.

Every candidate for this Degree must state in writing the special subject within the purview of the Faculty of Science, as set out in the Programme of the B.Sc. Examination, upon a knowledge of which he rests his qualification for the Doctorate; and with this statement he shall transmit an original Dissertation or Thesis (at least six copies), printed, type-written, or published in his own name, treating scientifically some special portion of the subject so stated, embodying the result of independent research, or showing evidence of his own work, whether conducted independently or under advice, and whether based on the discovery of new facts observed by himself, or of new relations of facts observed by others, or, generally, tending to the advancement of Science. Every candidate may further specify any printed contribution or contributions to the advancement of Science which he has at any time previously published. If the Dissertation or Thesis be approved by the Examiners, the candidate shall be required to present himself at the University upon such day or days within the first twenty-one days of June as may be notified to him, and shall, at the discretion of the Examiners, be further tested, either orally or practically, or by printed questions or by all of these methods, with reference both to the special subject selected by him and to the Thesis.

PRELIMINARY SCIENTIFIC (M.B.) EXAMINATION.

This Examination takes place twice in each year,—once, for Pass and Honours, commencing on the third

Monday in July; and once for Pass Candidates only, commencing on the third Monday in January.

No candidate shall be admitted to this Examination unless he shall have passed the Matriculation Examination. Not less than five weeks before the commencement of the Examination he must apply to the Registrar for a Form of Entry, which must be returned not less than four weeks before the Examination, accompanied with the candidate's fee.

The Fee for this examination is Five Pounds.

UNIVERSITY OF OXFORD.

Waynflete Professor of Chemistry—W. Odling, M.A., F.R.S.

Every Student must reside in one or other of the Colleges or Halls, or in licensed lodgings, for a period of three years. Students of Chemistry can obtain the degree of B.A. by passing preliminary examinations in Arts and in Science, and a final Honour examination in Chemistry. Chemistry may also be taken as part of the examination for a Pass degree. Graduates of other Universities suitably qualified can obtain the degree of Bachelor of Science after an approved course of study or research and two years' residence.

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Christ Church Laboratory.—A. Vernon Harcourt, F.R.S. Scholarships of about the value of £80 are obtainable at the majority of the colleges, by competitive examination in Natural Science.

More detailed information may be obtained from the Examination Statutes; the Student's Handbook to the University; and from the professors and college tutors.

UNIVERSITY OF CAMBRIDGE.

Professor of Chemistry—G. D. Liveing, M.A., F.R.S.

Jacksonian Professor of Natural and Experimental Philosophy—J. Dewar, M.A., F.R.S.

The Student must enter at one of the Colleges or Hostels, or as a Non-collegiate Student, and keep terms for three years by residence in the University. He must pass the previous examination in Classics and Mathematics, which may be done in the first or third term of residence, or, through the Oxford and Cambridge Schools Examination Board, or through the Senior Local Examinations, before commencing residence. He may then proceed to take a Degree in Arts, either continuing mathematical and classical study, and passing the ordinary examinations for B.A., or going out in one of the Honour Triposes.

The scholarships, ranging in value from £20 to £100 a year, are chiefly given for mathematical and classical proficiency. Scholarships, or Exhibitions, are given for Natural Science in King's, Trinity, St. John's, St. Peter's, Clare, Trinity Hall, Queen's, Jesus, Christ's, Sidney, Pembroke, Caius, and Downing Colleges; the dates of the examinations vary, but are always fully advertised.

The Chemical Laboratory of the University is open daily for the use of the Students. The Demonstrators attend daily to give instructions. A list of the lectures is published annually, in June, in a special number of the *Cambridge University Reporter*, which may be had from the Cambridge Warehouse, in Paternoster Row, or through any bookseller.

Non-collegiate Students are allowed to attend certain of the College Lectures and all the Professors' Lectures, and have the same University status and privileges as the other Students. Full particulars may be obtained by forwarding a stamped directed envelope to the Assistant Registrar, Cambridge, or from the *Cambridge University Calendar*.

UNIVERSITY OF DUBLIN.
TRINITY COLLEGE.

Professor of Chemistry—J. Emerson Reynolds, D.Sc., M.D., F.R.S.

Assistant Lecturer—Emil A. Werner, F.C.S., F.I.C.

Demonstrator—J. Percy Bailey, B.A.

The general Laboratories include working accommodation for 120 Students, and the Quantitative and Research Laboratories for about 40 Students. The Laboratories will open on the 1st of October. Lectures will commence about November 1st.

The Laboratories and the Lectures of the Professor of Chemistry can now be attended by Students who do not desire to reside in the University or proceed to its Degrees.

The full Course of General and Analytical Chemistry occupies three years, but a Student is free in his third year to devote most of his time to a special department of Pure or Technical Chemistry. Students can enter for any portion of the Course. The following Lectures are delivered:—

1. *Inorganic Chemistry and Chemical Philosophy*.—Elementary, first year; advanced, second year.
2. *Organic Chemistry*.—General, second year; advanced, third year.
3. *Metallurgy*.—A Course for Engineering and Technical Students.

The Laboratories are open every day from 10 to 5 o'clock (except Saturdays, when they close at 1 o'clock).

The Summer Course of Practical Chemistry for Medical Students begins during the first week in April and terminates with the first week in July.

The University of Dublin grants the Degree of Doctor of Science to graduates of Master's standing whose independent researches in any branch of Science are of sufficient merit.

KING'S COLLEGE.

(DIVISION OF ENGINEERING AND APPLIED SCIENCE).

Professor of Chemistry—J. M. Thomson, F.R.S., F.C.S.

Demonstrator of Practical Chemistry—Herbert Jackson, F.C.S.

Assistant Demonstrators—P. H. Kirkaldy, F.C.S., and W. H. Sodeau, B.Sc.

The Academical Year consists of Three terms. The days fixed for the Admission of New Students in the Academical Year 1897-98 are September 30, January 13, and April 27.

Students of the First Year are admitted to the Course of Theoretical and Applied Chemistry. The Course commences with a view of the conditions suitable for the production of Chemical Phenomena, after which the laws of Chemical Attraction are discussed, and the Non-metallic Elements and their principal compounds are described. The Metals and their principal compounds are next examined, care being taken to point out the applications of the Science to the Arts; and the processes of the different Manufactures and of Domestic Economy are explained and illustrated. Examinations of the Class, both *visà voce* and by written papers, are held at intervals during the course at the usual Lecture hour.

Second Year.—Students attend in the Laboratory twice a week, and they go through a course of Manipulation in the most important operations of Chemistry, including the first steps of Analysis. Any Student of this Division may be admitted to this Class at any period of his study on payment of an extra fee.

Experimental and Analytical Chemistry in the Laboratory.—The object of this Class is to afford to Students who are desirous of acquiring a knowledge of analysis, or of prosecuting original research, an opportunity of doing so under the superintendence of the Professor and Demonstrator; Students may enter, upon payment of extra fees, at any time except during the vacation, and for a period of one, three, six, or nine months, as may best suit their convenience. The laboratory hours are from ten till

four daily, except Saturday, on which day the hours are from ten till one.

In addition to the Laboratory Fee, each Student defrays the expenses of his own experiments. The amount of this expense, which is comparatively trifling, is entirely under his own control.

Special hours and fees are arranged for the convenience of such Third Year Students as wish to study Analytical Chemistry.

Fees.—Chemistry per term, £3 3s. 6d.; per ann., £8 8s. 6d.; Practical Chemistry per term, £4 4s. 6d.; per ann., £10 10s. 6d.; Experimental and Analytical Chemistry—Daily attendance: One month, £4 4s.; Three months, £10 10s.; Six months, £18 18s.; Nine months, £26 5s. Three days a week: One month, £2 12s. 6d.; Three mos., £6 6s.; Six mos., £11 11s.; Nine mos., £15 15s.

METALLURGY.

Professor—A. K. Huntington, F.I.C., F.C.S., &c.

The following subjects are treated of in the Lectures: The Selection and Economic Preparation of Fuel and of Refractory Materials; the methods by which metals are obtained from their ores, and the means by which they are rendered suitable for the various requirements of the Arts.

Particular attention is paid to the study of the Nature and Properties of Metals and Alloys available for Constructive Purposes.

In the Metallurgical Laboratory, which is always open during College hours, the relation between the Chemical Composition of Metals and their Mechanical Properties may be studied by the aid of Testing Machinery.

PHOTOGRAPHY.

Lecturer—Prof. J. M. Thomson, F.R.S., F.C.S.

In addition to the regular College Course in Photography occasional classes may be formed. For further particulars application should be made to Prof. Thomson.

EVENING CLASSES.

Classes for Evening Instruction in various subjects are held during the months from October to March, inclusive, and during the months of April, May, and June.

UNIVERSITY COLLEGE.

FACULTY OF SCIENCE.

Professor—William Ramsay, Ph.D., F.R.S.

Assistants—Morris Travers, B.Sc., Alexander Kellas, B.Sc., and J. W. Walker, M.A., Ph.D.

The Session is divided into three Terms, as follows, all the dates being inclusive:—

First Term, from Tuesday, October 5th, until Friday, December 17th;

Second Term, from Tuesday, January 11th, 1898, till Friday, April 1st;

Third Term, from Tuesday, April 26th, till Tuesday, July 5th. Class Examinations begin on June 22nd.

Junior Courses of Inorganic Chemistry.

First Term: Tuesday, Thursday, and Saturday at 10. Second and Third Terms: Tuesday, Thursday, and Saturday. Fee:—£4 4s.

These Courses will each consist of about thirty lessons, partly theoretical and partly practical, on the non-metallic elements. Frequent exercises will be given.

Senior Course of Inorganic Chemistry.

First and Second Terms: The Class meets four times a week, on Mondays, Wednesdays, Fridays, and Saturdays, at 9, for Lectures. Examinations, and Exercises.

Fees:—For the Course, £7 7s.; Perpetual, £9 9s.; for the First or Second Terms, £4 4s.

This Course and the Practical Class cover the subject as prescribed for the Preliminary Scientific (M.B.) and Int. Examination in Science of the University of London.

For the Preliminary Scientific Examination Students who take the three subjects for that examination in July attend during the First and Second Terms.

Advanced Course of Chemistry.

Second and Third Terms.—The class meets twice a

week, on Tuesdays and Thursdays, at 9, beginning on January 13. The hour will be altered by special arrangement with the class if necessary.

Fee:—For the Course, £3 3s.; for a Term, £2 2s.

This Course will be found suitable for those about to proceed to graduation as Bachelor of Science in London University, and to those who intend to choose Chemistry as a profession. Such students should also work in the Laboratory during as many hours as they can spare.

Organic Chemistry.

Tuesday, Thursday, and Saturday, at 9, in the First Term; Tuesday, Thursday, and Saturday, at 10, in the Second Term; and Tuesday and Thursday at 9, and Saturday at 11, in the Third Term. The hour of meeting will be altered should the class desire it.

This Course of Organic Chemistry is intended for those who are studying the subject from a scientific standpoint. Candidates for Honours at the Int.M.B. are, however, recommended to attend this Course besides the Special Summer Course.

The Course includes the subjects required at the B.Sc. Examination, Pass and Honours; but no previous acquaintance with Organic Chemistry will be expected of those joining the Class.

Fee:—For the Course, £6 6s.; for the Second and Third Terms, £4 14s. 6d.; for a Term, £2 12s. 6d.; for a Second Course, £3 3s.

Practical Classes.

Practical Classes in Inorganic and Organic Chemistry are conducted by the Assistants.

Analytical and Practical Chemistry.

The Laboratory is open daily from 9 a.m. to 4 p.m., Saturdays excepted, from October until the middle of July, with a short recess at Christmas and at Easter.

Fees: for the Session, £26 5s.; six months, £18 18s.; three months, £10 10s.; one month, £4 4s.

Three specified days a week:—for the Session, £15 15s.; six months, £11 11s.; three months, £6 6s.; one month, £2 12s. 6d., exclusive of expense of materials. Students may enter at any period of the Session.

The Laboratory Course includes the Practical Chemistry required at the following Examinations of the University of London:—Prel. Sci. (M.B.), Intermediate M.B., Intermediate Science, B.Sc.

Students who wish to attend the Lectures on Chemical Technology may acquire here the requisite knowledge of Practical Chemistry and Analysis.

When accompanied by, or preceded by, attendance on the Lectures on Inorganic and Organic Chemistry, the Laboratory Course qualifies Students in the application of Chemistry to Manufactures, Metallurgy, Medicine, or Agriculture, &c.

There is also a Chemical Library containing the chief Journals and Standard Works on Chemistry.

Certificates of Honour are granted to competent Students on the work done during the Session. The Tuffnell Scholarship (£100 for two years) will also be competed for in the Session 1897-98; also the Cloth-worker's Scholarship of £30.

ROYAL COLLEGE OF SCIENCE AND ROYAL SCHOOL OF MINES.

Professor—W. A. Tilden, D.Sc., F.R.S.

Assistant Professor—W. P. Wynne, D.Sc., F.R.S.

Demonstrators—H. Chapman Jones and J. W. Rodger, A.R.C.S.

Assistants—G. S. Newth, A. Eiloart, Ph.D., B.Sc., and M. O. Forster, Ph.D.

The Royal College of Science at South Kensington is intended, primarily, for the instruction of teachers, and of students of the industrial classes selected by competition in the examinations of the Science and Art Department. The Royal School of Mines is incorporated with the Royal College of Science. Students entering for the Associateship of the Royal School of Mines obtain their general scientific

training in the Royal College of Science. The instruction in the Royal College of Science is arranged in such a manner as to give the Students a thorough training in the general principles of Science, followed by advanced instruction in one or more special branches of Science. The Associateship is granted in certain divisions or lines of study. Students who go through any one of the prescribed courses of instruction and pass the necessary Examinations receive a Certificate of Associateship of the Royal College of Science, or of the Royal School of Mines. Students who are not candidates for the Associateship are permitted to enter as occasional students in one or more special branches of science, and on passing the examination receive a Certificate to that effect. The Associateship of the Royal College of Science is given in one or more of the following divisions:—Mechanics, Physics, Chemistry, Biology, Geology, and Agriculture, and the Associateship of the Royal School of Mines in Metallurgy and Mining.

The course of instruction, which lasts for three years, is the same for all the divisions during the first year, after which it is specialised in accordance with the Scheme detailed in the Prospectus of the School.

The Session is divided into two Terms. The first Term begins on the 7th of October and ends about the middle of February. The second Term begins in the middle of February and ends about the middle of June.

Examinations are held at the end of each course of instruction and at such other periods as may be found necessary. On the results of these examinations the successful candidates are arranged in two classes, first and second. There are also "Honours" examinations for the subjects of the third year, the successful candidates being placed in order of merit. A student obtains the Associateship who passes in all the subjects of the first two years and in the third year those of the special division he selects for his Associateship. A student who goes through the prescribed course of instruction in any subject and passes the final examination in it receives a certificate to that effect.

Students who do not wish to attend the lectures are admitted for short periods to the laboratories, at the discretion of the Professors. The fees for the laboratories are £4 per month.

Students not entering for the Associateship are admitted to any particular course of study, so far as there is room, on payment of the fees shown in the following table:—

	Lectures.	Laboratory.
Chemistry	3	13
Physics	5	12
Biology with Botany	5	12
Geology with Mineralogy	4	8
Mechanics	4	6
Metallurgy	2	13
Mining	4	
Astronomical Physics	2	3

Agricultural Chemistry, per term, £13. Mathematics and Mechanical Drawing, £3 per term. Model and Free-hand Drawing, £1 per term. Descriptive Geometry, £3 per session. Mine Surveying, £10.

The fees for the first two years amount to about £75, and for the remainder of the course for the Associateship they vary from £30 to about £40.

Both the private and the State-aided students are required to furnish themselves with certain instruments and apparatus before the commencement of the courses. These are enumerated in the syllabuses of the several subjects.

Officers of the Army, Navy, and Civil Service, recommended by their respective Departments, are admitted to the Lectures and Laboratories at half fees.

Associates of the Royal College of Science or of the Royal School of Mines have the privilege of free admission to the Library and to all the courses of lectures.

Bona fide teachers qualified to earn payments for teaching Science according to the rule of the Science and

Art Directorate may obtain permission to attend free any course of lectures.

Several valuable Exhibitions, Scholarships, and Prizes are attached to the studentship.

Summer Courses for Teachers.—Short courses of instruction are given annually, about July, in different branches of science for the benefit of teachers of science schools in the country. The courses last three weeks. About 250 teachers are admitted to them, and they receive third class railway fare to and from South Kensington, and a bonus towards their incidental expenses of £3 each. (See Science and Art Directorate.)

Working Men's Lectures.—Notification of these will be given in the newspapers.

THE SCHOOL OF THE PHARMACEUTICAL SOCIETY OF GREAT BRITAIN.

The Fifty-sixth Session will commence on Monday, October 4th, 1897.

Professors.—Chemistry, J. Norman Collie, Ph.D., F.R.S.; Botany, J. Reynolds Green, Sc.D., F.R.S., F.L.S.; Materia Medica and Pharmacy, Henry G. Greenish, F.I.C., F.L.S. (Dean).

A Course of Lectures on Physical, Inorganic, and Elementary Organic Chemistry commences in October and terminates at the end of June. An Advanced Course of Lectures begins in October and extends to the end of March. These Lectures are adapted to the requirements of Pharmaceutical and Medical Students, and also those who are proceeding to degrees at the University of London, or who are preparing for the examinations of the Institute of Chemistry.

Entries may be made for single classes. Certificates of attendance at the two Courses of Lectures on Chemistry and at the Chemical Laboratories are accepted as evidence of chemical training by the Institute of Chemistry in connection with the Examinations for the Associateship, and also by the conjoint Board of the Royal Colleges of Physicians and Surgeons, as well as by other examining bodies.

Prospectuses and further information may be obtained from Mr. Richard Bremridge, Secretary and Registrar, 17, Bloomsbury Square, London, W.C.

UNIVERSITY COLLEGE OF WALES, ABERYSTWYTH.

UNIVERSITY OF WALES.

Professor—H. Ll. Snape, D.Sc. (Lond.), Ph.D. (Göttingen), F.I.C.

Assistant Lecturer and Demonstrator—A. W. Warrington, M.Sc. (Vic.), F.I.C.

Lecturer in Agricultural Chemistry—J. Alan Murray, B.Sc. (Edin.).

The College is open to male and female students above the age of sixteen years. The Session commences on Tuesday, October 5, on which day all Students will be expected to meet the Professors in the Examination Hall of the College.

Lecture Courses.—(1) Matriculation Course; three lectures weekly during the Michaelmas and two weekly during the Lent and Easter Terms. (2) Intermediate Science Pass Course; four lectures weekly during the Lent and Easter Terms. (3 and 4) B.Sc. Courses; A, three lectures weekly on Organic Chemistry; B, two lectures weekly on Chemical Theory. (Courses A and B will generally be given in alternate Sessions; for 1897-8, Course A.) (5 and 6) Courses in Agricultural Chemistry. For students in their first year, 3 lectures, and for those in their 2nd year, 2 lectures weekly throughout the Session.

Laboratory Courses.—The Laboratory is open daily from 10 a.m. to 1 p.m., and from 2.15 to 5 p.m., except on Wednesdays and Saturdays. Classes for the Systematic Study of Qualitative and Quantitative Analysis will be formed, and Special Courses will be

arranged for those who intend to follow Medicine or Pharmacy, or any one particular branch of Applied Chemistry, always provided that such Students possess the requisite knowledge of Theoretical Chemistry. The hours will be arranged, as far as possible, to suit the requirements of the individual Student.

The College is recognised by the Royal University of Ireland, and by the Colleges of Physicians and Surgeons of England, Scotland, and Ireland as an institution at which the instruction necessary for their respective Diplomas in Medicine, in Chemistry, Physics, and Biology may be given. One year for graduation in Medicine and two years for graduation in Science may be spent at Aberystwyth.

Fees.—The Fee for the whole Session, if paid in advance, is £10; if paid by Single Terms, for the first term of attendance in each Session, £4; for the second term, £3 10s.; for the third term, £3. These composition fees enable the Student to attend any or all the Classes of the College, with the exception that a small extra fee is charged for Laboratory Instruction. Thus, for Practical Chemistry, the additional fee is, for six hours' work per week, 10s. per term, and for twelve hours, 20s. per term. The fees for those who desire to spend several days weekly in the laboratory may be learned on application to the Registrar. Fee for a single Lecture Course £1 per term.

Scholarships and Exhibitions varying in value from £10 to £40 per annum will be offered for competition at examinations which commence on September 21, and exhibitions are awarded at the end of the Session on the results of the class examinations.

The Chemical Laboratories in connection with this College have been recently built, and are fitted with every convenience for the prosecution of chemical studies.

Intending Students requiring further information are recommended to write to the Registrar for a copy either of the General Prospectus or of one of the Special Prospectuses issued for the Agricultural and Normal Departments.

UNIVERSITY COLLEGE OF NORTH WALES, BANGOR.

A CONSTITUENT COLLEGE OF THE UNIVERSITY OF WALES.

Chemistry.—Professor, James J. Dobbie, M.A., D.Sc. Demonstrator, Fred. Marsden, Ph.D., B.Sc. Assistant Lecturer in Agricultural Chemistry, F. V. Dutton.

Physics.—Professor, Andrew Gray, M.A., LL.D., F.R.S.

The Session opens October 5th, 1897. All regular classes are open to men and women students above the age of 16 years. The following Courses of Lectures will be given.

Matriculation Course.—Subjects: Those prescribed for the Matriculation Examination of the University of Wales. Fee for the Term £2 2s. A class for revision of Matriculation Work will be held during the Summer Term. Fee for the Term, £1 1s.

Intermediate Course.—Inorganic Chemistry and Elementary Physical Chemistry. Fee for the Term £2 2s.

B.Sc. Course.—Advanced Inorganic Chemistry. Fee for the Session, £3 3s.

Medical Course.—Fee, £4 4s.

Agricultural Chemistry.—Fee, £2 2s.

Laboratory Courses.—The laboratory is open on five days of the week from 10 a.m. to 4 p.m. for instruction in Chemical Analysis and in the Application of Chemistry to Medicine and the Industrial Arts. Fees: six hours per week, £1 1s. per Term; twelve hours, £2 2s.; eighteen hours, £3 3s.; twenty-four hours, £4 4s. Composition Fee for all Laboratory Classes of the Intermediate Science Course taken in one year, £4 4s.

The Chemistry, Botany, Zoology, and Physics Courses are recognised for Medical graduation in the Universities of Edinburgh and Glasgow, and students can make one *Annus medicus* at the college. The Science Courses are

recognised for part of the science degree course of the University of Edinburgh.

UNIVERSITY COLLEGE OF SOUTH WALES AND MONMOUTHSHIRE, CARDIFF.

Professor—C. M. Thompson, M.A., D.Sc., F.C.S.

Demonstrators—E. P. Perman, D.Sc., F.C.S., and A. A. Read, F.I.C., F.C.S.

The Session commences October 4th, and terminates on June 24th, and is divided into three terms.

The Junior Course (delivered during the Michaelmas term only) consists of about 50 lectures, and will cover the subjects prescribed for the Matriculation examinations of the University of Wales and the University of London. Fee, £2 2s. A revision class is held in the Summer term.

The Intermediate Course consists of about 80 lectures held during the Lent and Summer terms in continuation of the Junior Course, and is the qualifying course for the Intermediate Examination of the University of Wales. Together with laboratory practice, it will cover the subjects required for the Intermediate Examination in Science and the Prel. Sci. (M.B.) Examination of the University of London. Fee, £4 4s.

The Senior Course consists of some 90 lectures on Organic Chemistry; Fee, £3 3s.

A course of 20 lectures on Qualitative Analysis and a short course on Organic Chemistry will also be given.

The following lectures on Metallurgy will be given by Mr. Read:—10 lectures on Fuel; Fee, 10s. 6d. 20 lectures on General Metallurgy; Fee, £1 1s. 30 lectures on the Manufacture of Iron and Steel; Fee, £1 1s. A practical course on Iron and Steel Analysis will also be held, and practical instruction in Dry Assaying will be given in the Metallurgical Laboratory, which is fitted with the necessary furnaces and apparatus.

In the laboratory each student works independently, so that the course of study may be adapted to the requirements of the individual. Hours, 9 to 1 and 2 to 5; Saturday, 9 to 1. Fees—Six hours per week, £3 3s. per session; twelve hours, £2 2s. per term; eighteen hours, £3 3s. per term; twenty-four hours £4 4s. per term.

Registered medical students can prepare for the Intermediate M.B. Examination of the University of London, and spend three out of their five years of medical study in Cardiff. Medical students wishing to graduate at a Scottish University, or preparing for a Conjoint Board Surgical and Medical Diploma, or for the Diploma of the Society of Apothecaries, can spend two years in Cardiff. For further information see the prospectus of the Faculty of Medicine, which may be obtained from the Registrar.

The College is recognised as an institution at which two years of the course for the degree of Bachelor of Science of the University of Edinburgh may be spent.

Students by making a payment of £10 at the commencement of each session may compound for all lecture fees for the whole session. Laboratory fees are not included in the composition fee, but Students preparing for the Science Examinations of the University of Wales and of the University of London may, by making a payment of £13 13s. at the commencement of each Session, compound for both Lecture and Laboratory Fees during the Session.

At the entrance examination in September, and the annual examination in June, several scholarships and exhibitions are awarded. Great importance is attached to special excellence in one subject.

The College Prospectus, and also further information as to scholarships, may be obtained from the Registrar.

A Hall of Residence for Women Students is attached to the College.

UNIVERSITY COLLEGE, BRISTOL.

Professor of Chemistry—Sydney Young, D.Sc., F.R.S.

Lecturer—Francis E. Francis, B.Sc., Ph.D.

The session 1897-98 will begin on October 5th. Lectures and classes are held every day and evening throughout

the Session. In the Chemical Department lectures and classes are given in all branches of theoretical chemistry, and instruction in practical chemistry is given daily in the chemical laboratory. The department of experimental physics includes various courses of lectures arranged progressively, and practical instruction is given in the physical and electrical laboratories. The Department of Engineering and the Constructive Professions is designed to afford a thorough scientific education to students intending to become engineers, or to enter any of the allied professions, and to supplement the ordinary professional training by systematic technical teaching. This department includes courses specially arranged for students intending to become civil, mechanical, electrical, or mining engineers, surveyors, or architects. Those who attend the mechanical engineering course enter engineering works during the six summer months, and, in accordance with this scheme, various manufacturing engineers in the neighbourhood have consented to receive students of the College into their offices and workshops as articulated pupils at reduced terms. Medical education is provided by the Faculty of Medicine of the College. Several Scholarships are tenable at the College. Full information may be obtained from the Secretary.

DAY LECTURES.

Inorganic Chemistry.

The Courses treat of the principles of Chemistry, and of the Chemistry of the Non-Metals and Metals.

Junior Course.—Two Lectures a week will be given during the First and Second Terms. Fee, £3 3s.

Special Course.—A special course of Lectures is also given to Engineering Students.

Senior Course.—Three Lectures a week will be given throughout the Session. Fee, £5 5s. There will be tutorial classes in connection with the Junior and Senior Courses.

Advanced Course.—One Lecture a week will be given throughout the Session. Fee, £2 12s. 6d.

Organic Chemistry.

This Course will relate to the more important groups of the Compounds of Carbon.

Two Lectures a week will be given during the Second Term, and three Lectures a week during the Third Term. Fee, £3 3s. An advanced course of lectures will also be given one day a week during the session. Fee, £2 12s. 6d.

Practical Chemistry.—Laboratory Instruction.

The Laboratory will be open daily from 10 a.m. to 5 p.m., except on Saturdays, when it will be closed. Instruction will be given in the Laboratory in all branches of Practical Chemistry, including Qualitative and Quantitative Inorganic and Organic Analysis, the preparation of Chemical Products, and Inorganic and Organic Research. Special facilities will be afforded to those who desire to study Practical Chemistry as applied to the different processes employed in the Arts and Manufactures. Fees in Guineas—

	5 Days a Week.	4 Days a Week.	3 Days a Week.	2 Days a Week.	1 Day a Week.
Per Session ..	15	12½	10	7½	5
„ Two Terms ..	11	9	7½	5½	3½
„ One Term ..	7	6	4½	3½	2½

Students may arrange to divide their days of laboratory work into half-days.

Chemical Scholarship.—Among others, a Chemical Scholarship of £25 is offered for competition.

EVENING LECTURES.

Two courses of Lectures will be delivered during the First and Second Terms; they will be devoted to the consideration of the general Principles of Chemistry and Chemical Physics and the Chemistry of Non-Metallic and Metallic Elements. Special attention will be paid throughout to those products which have a practical application in the Arts and Manufactures. Fee for each course, 7s. 6d.

Practical Chemistry.—Laboratory Instruction.—The

Laboratory will be open on Tuesday and Wednesday evenings from 7 till 9. Instruction will be given in Qualitative and Quantitative Analysis, and in the Preparation of Chemical Products. Fees:—(Two Terms) Two Evenings, 25s.; One Evening, 15s. (One Term) Two Evenings, 15s.; One Evening, 10s 6d.

University College, Bristol, has been approved by the Council of the Institute of Chemistry as a College at which all the subjects required for the admission of Associates to the Institute are taught.

The Calendar of the College, price 1s. (post-free, 1s. 4d.), containing detailed information of the various Courses, may be obtained on application to the Secretary.

MASON COLLEGE, BIRMINGHAM.

Professor—Percy F. Frankland, Ph.D., B.Sc., F.R.S.

Assistant Lecturer—C. F. Baker, Ph.D., B.Sc.

Demonstrator—W. R. Innes, Ph.D., M.Sc.

The Session will be opened on September 30th, 1897.

Elementary Course.

Forty Lectures adapted to the requirements of beginners will be given in the Winter and Spring Terms. Lecture days—Wednesdays and Fridays at 11.30.

Persons entirely unacquainted with Chemistry are recommended to attend this Course before entering for the General Course. Candidates for the Matriculation Examination of the University of London also are advised to attend this Course.

General Course.

The General Course of Lectures on Chemistry will be found useful by Students who are afterwards to become Engineers, Architects, Builders, Brewers, or Manufacturers (such as Metallurgists, Alkali, Soap, Manure, Glass, or Cement Makers, Bleachers and Dyers, &c.)

Students preparing for the Intermediate Examination in Science and Preliminary Scientific (M.B.) Examination of the University of London should attend the Lectures on Inorganic Chemistry (Winter and Spring Terms).

Candidates for Intermediate Examinations in Medicine will in general require only that part of the course (Summer Term) which relates to Organic Chemistry.

The full course, extending over three terms, will also satisfy the requirements of Students preparing for the Associateship of the Institute of Chemistry, so far as attendance at lectures on General and Theoretical Chemistry is concerned.

1. From October to March (Winter and Spring Terms). About eighty lectures on Inorganic Chemistry and Chemical Philosophy will be given on Mondays, Tuesdays, Wednesdays, and Thursdays from October to December, and on Mondays, Tuesdays, and Wednesdays from January to March, at 9.30 a.m. A Tutorial Class is held in connection with this Course once a week throughout the Session. Fee, £5 5s. for the course.

2. April to June (Summer Term). About thirty lectures will be given on Elementary Organic Chemistry, or the chemistry of the most important series of carbon compounds. This course will include all the subjects required for the Intermediate Examination in Medicine of the University of London. Lecture Days—Monday, Wednesday, and Friday at 12 noon. Fee, £1 11s. 6d.

The General Course (including Inorganic and Organic lectures) qualifies for graduation in the medical faculties of the universities of Edinburgh, Glasgow, Aberdeen, and Durham.

Special Courses of Lectures and of Laboratory Instruction are given for Medical Students preparing for the Conjoint Board Examinations.

Advanced Course.

An Advanced Course for the study of Theoretical Chemistry and those parts of the subject which are required for the degree of B.Sc. in the University of London will meet twice a week. Fee for the session £3 3s.

Laboratory Practice.

The College Laboratory is open daily from 9.30 to 5, except on Saturdays, when it is closed at 1 p.m.

Candidates for Intermediate Examination in Science, Preliminary Scientific (M.B.), B.Sc., and Intermediate Examination in Medicine of the University of London, may obtain in the Laboratory of the College the instruction necessary. The three months Course of Practical Chemistry for the B.Sc., Edinburgh, in the department of Public Health, may be taken in the Mason College Laboratory. Fees:—

	All day.	Three hours per day.
One Term	7 guineas	4½ guineas.
Two Terms	13 "	8½ "
Three Terms	18 "	12 "

A Course of short demonstrations and exercises is given by the Professor or one of his Assistants once a week. All first-year Students are required to attend, unless exempted for special reasons by the Professor. No Fee.

Metallurgy.

Three Courses of Ten Lectures will be given on the Principles and Practice of Metallurgy. Fee, 10s. 6d. for each of the first two courses, and for each of the two sections of the third course. A more advanced course of about sixty lectures upon selected subjects is also given.

There is a separate laboratory for metallurgical students in which provision is made for instruction in assaying, &c.

Evening Classes.

Special Courses of Evening Lectures are arranged during the Winter and Spring Terms of each session. The subjects are treated in a less technical manner and the fees are nominal.

Scholarships.

Priestley Scholarships.—Three Open Scholarships in Chemistry of the value of £100 each are awarded annually in September.

Bowen Scholarship.—One Open Scholarship in Metallurgy of the value of £100 is awarded annually in September.

Forster Research Scholarship.—A Scholarship of the value of £50 is annually awarded.

For particulars apply to the Registrar.

Excursions.

During previous Sessions permission has been obtained to visit some of the great factories in or near Birmingham, in which chemical and metallurgical industries are carried on. Students have thus had most valuable opportunities of gaining a practical acquaintance with some branches of Applied Science. The privilege thus courteously granted by several manufacturers will, it is hoped, be enjoyed in every future Session. The excursions will be conducted by the Professor or Lecturer.

BRADFORD TECHNICAL COLLEGE.

CHEMISTRY AND DYING DEPARTMENT.

Head Master—W. M. Gardner, F.C.S.

Demonstrator and Lecturer on Geology—A. B. Knaggs, F.C.S.

Lecturer on Botany and Biology—William West, F.L.S. The school year is divided into three terms. The Session commences on September 14th and terminates on July 17th. The course of instruction extends over two years, and embraces Lecture Courses on Inorganic and Organic Chemistry, the technology of the textile fibres, mordants, natural and artificial colouring matters, technical analysis, and laboratory practice in analytical chemistry, chemical preparations, and dyeing. Fee, £5 per Term, or £13 per Session.

During the first and second terms Evening Classes are held for the benefit of persons engaged during the day and for pharmaceutical students.

ROYAL AGRICULTURAL COLLEGE,
CIRENCESTER.

CHEMICAL DEPARTMENT.

Professor—Prof. E. Kinch, F.C.S., F.I.C.*Assistants*—Cecil C. Duncan, F.I.C., and W. James.

Systematic courses of Lectures are given on the various branches of Chemistry in its relation to Agriculture, illustrated by experiments, and by the collections in the College Museum. They comprise the laws of Chemical Combination and the general Chemistry of mineral bodies, and of the more frequently occurring bodies of organic origin, with the relationships of their leading groups; and, finally, the applications to practical operations of the Chemistry of the atmosphere, of soils and manures, of vegetation and stock feeding, and of the processes and products of the dairy.

In the Laboratory practical instruction is given in the construction and use of apparatus and in Chemical manipulation and analysis, both qualitative and quantitative. After studying the simple operations and the properties of the commonly occurring substances, the Students are taught to analyse a series of compounds, and apply the knowledge thus obtained to the analysis of manures, soils, waters, feeding stuffs, dairy products, and other substances met with in the ordinary course of Agricultural practice. Chemico-agricultural researches are undertaken by the senior Students under the direction of the Professor and his Assistants.

VICTORIA UNIVERSITY.
THE YORKSHIRE COLLEGE, LEEDS.*Professor of Chemistry*—Arthur Smithells, B.Sc. Lond., F.I.C.*Lecturer in Organic Chemistry*—Julius B. Cohen, Ph.D., F.I.C.*Assistant Lecturer and Demonstrator*—Herbert Ingle, F.I.C.*Demonstrator*—T. S. Patterson, Ph.D.

The Session begins October 5, 1897.

Lecture Courses.

1. General Course of Chemistry.—Monday, Wednesday, and Friday, at 11.30 a.m., from October to the end of the second term, and during part of the third term. Fee for the Course, £4 4s.

2. Inorganic Chemistry.—First year Honours Course, Non-metals. Monday, Wednesday, and Friday, at 9.30 a.m. Fee, £3 13s. 6d.

3. Inorganic Chemistry.—Second year Honours Course, Metals. Tuesday, Thursday, and Saturday at 9.30 a.m. Fee, £3 13s. 6d.

4. Organic Chemistry.—Tuesday, Thursday, and Saturday at 12 noon. Fee £3 13s. 6d.

5. Organic Chemistry Honours Course.—Wednesday and Friday at 12 noon. Fee, £2 12s. 6d.

6. Theoretical Chemistry.—Advanced Course. Tuesdays and Thursdays at 9.30 a.m. Fee, £2 12s. 6d.

7. Chemistry as Applied to Coal Mining.—Tuesday during the First Term, at 4 p.m.

8. Chemistry for Teachers.—Saturdays from 9.30 to 12.30 in the first and second terms. Fee, £4 4s.

Laboratory Courses.

The College Laboratory will be open daily from 9 a.m. to 1 p.m., and from 2 to 5 p.m., except on Saturdays, when it will close at 1 p.m.

Fees for the Session—Students working six days per week, £21; five, £18 18s.; four, £16 16s.; three, £13 13s.

Class in Practical Chemistry, Saturday mornings, from 9.30 to 12.30. Fee £1 11s. 6d.

Practical Chemistry for Medical Students.—Tuesdays, 9.30 to 11.30 October to end of December; Thursdays, 2 to 4 from January to end of March.

Practical Course in Sanitary Chemistry.—Tuesdays and Thursdays from 2 to 5 p.m., from January to March. Fee, £5 5s.

Practical Organic Chemistry for Medical Students.—

Tuesdays and Thursdays from 10 to 12 during the Third Term. Fee, £2 2s.

Evening Class.

A Course of twenty Lectures by Mr. Ingle, on the Chemistry of Photography will begin during the first and second Terms. Fee, 10s. 6d.

Dyeing Department.

Professor—J. J. Hummel, F.I.C.*Lecturer and Research Assistant*—A. G. Perkin, F.R.S.E.*Assistant Lecturer*—R. B. Brown.

This Course extends over a period of three years, and is intended for those who wish to obtain a full scientific and practical education in the art of dyeing. It is suitable for those who purpose in the future to take any part in the direction of the operations of dyeing or printing of textile fabrics, e.g., the sons of manufacturers, calico printers, managers, master dyers, &c.

Leather Industries Department.

Professor—H. R. Proßer, F.I.C.

The full Course, which extends over a period of three years, is suitable to all who intend to become Technical Chemists in the Leather Industry, or managers of important works, and is recommended to sons of tanners. The Course includes instruction in chemistry, engineering, leather manufacture, and practical work in the Leather Industries Laboratory.

Agricultural Department.

Professor—James Clark, M.A., Ph.D.

The full Course occupies two years, and includes instruction in chemistry, physics, botany, engineering and surveying, and the principles of agriculture, as well as practical work in the various laboratories and out-door agriculture.

Research Students are admitted to the College Laboratories on reduced terms.

Several valuable Scholarships are at the disposal of the College, viz., the Cavendish, Salt, Akroyd, Brown, Emaley, Craven, and Clothworkers' Scholarships, and the Leighton Trustees' Exhibition, and one of the 1851 Exhibition Scholarships. The West Riding County Council Scholarships are tenable at the Yorkshire College.

UNIVERSITY COLLEGE, LIVERPOOL.

Professor—J. Campbell Brown, D.Sc.*Lecturer on Organic Chemistry*—C. A. Kohn, B.Sc., Ph.D.*Lecturer on Metallurgy*—T. L. Bailey, Ph.D.*Demonstrators and Assistant Lecturers*—T. L. Bailey, Ph.D., C. A. Kohn, B.Sc., Ph.D., and A. W. Titherley, M.Sc., Ph.D.*Assistant*—H. H. Froyssell.

The Session commences October 4th.

The William Gossage Chemical Laboratory has been completed and opened for Advanced Students, the Metallurgical Laboratory has been enlarged, and new Gas Analysis and Electro-Chemical rooms have been added, with a third Lecture Room.

The Classes meet the requirements of candidates for the Ordinary B.Sc. Degree, for Chemistry Honours, or for the M.Sc. or D.Sc. Degree in Victoria University; for Degrees in Medicine of Victoria, London, and Edinburgh; for the Pharmaceutical Diplomas; for a special Technological Certificate of University College; and for those studying Chemistry as a preparation for professional, technical, or commercial life. The Classes qualify for the Fellowship of the Institute of Chemistry of Great Britain and Ireland, and other Examination Boards.

Lecture Courses.

General Elementary Course on the principal non-metallic elements and the most important metals, the principles of Chemical Philosophy, and an introductory sketch of Organic Chemistry. Three Terms. Fee, £4.

Engineer's Course of Lectures with Practical Class. Two Terms. Fee, £4.

Dental Course, Lectures and Practical. Fee, £5 5s.
Course A.—Non-metals. Fee, £3 10s.
Course B.—Metals. Fee, £3 10s.
Course C.—Organic Chemistry. Fee, £3 10s.
Course H.—Special Organic Subjects. Fee, £1.
Course D.—Physical Chemistry. One Term. Fee, £1.
Course E.—History of Chemistry and of the Development of Modern Chemical Philosophy. Three Terms. Fee, £2.

Courses F.—Technological Chemistry and Metallurgy: Lectures on Technology are given in connection with Laboratory work at hours to be arranged. The subjects are varied in different years. (1) Alkali and Allied Manufactures. (2) Copper, Iron, and Steel. (3) Lead, Silver and Gold, Aluminium, and other Metals. (4) Distillation of Coal and Tar Industries. (5) Fuel and Gas. (6) Chemistry Applied to Sanitation. (7) Technical Gas Analysis. Three terms. Fee, each course £1 10s.

Practical Classes.

(1) Junior. (2) Intermediate: Qualitative Analysis of Inorganic Substances and of some of the more common Organic Substances. (3) Revision Class. (4) Senior: Practical Organic (Advanced Medical Class). (5) Practical Exercises on Technology, Pharmaceutical Chemistry, Saitany subjects, Examination of Water and Air, of Animal Secretions, Urinary Deposits, Calculi, and Poisons. (6) Quantitative Class: Course arranged to suit the requirements of the London University B.Sc. Examinations, Pass and Honours, and for Intermediate M.B. Honours.

Chemical Laboratory.

The Chemical Laboratories provide accommodation for every kind of chemical and metallurgical work.

The William Gossage Laboratory, opened in 1897, consists of a large and well-fitted general Laboratory for advanced Students, a new gas analysis room, an additional lecture-room for Metallurgy and other classes, and an addition to the Research Laboratory. New stores for students' apparatus and chemicals have also been built and placed in charge of a skilled dealer.

Students desirous of gaining a thorough theoretical and practical acquaintance with Technical Chemistry, or who intend to adopt Chemical work as a profession, must devote three or four years to special study, for which a full curriculum is provided.

TABLE OF FEES.

Per Week.	One Term, Three Months.	Three Terms, One Session.
One day	£4	£7
Two days	6	10
Three days	8	13
Four days	9	16 10s.
Whole week	10 10s.	21

Pharmaceutical Course (see special syllabus).

Technological Curriculum.

Preliminary Year.—Chemistry, the Elementary Course. Practical Classes 1 and 2. Mathematics, or Mechanics, or Physics. Elementary Engineering, Drawing, and Design (in this or one of the following years). German. Or, the Victoria Preliminary Course and Examination may be taken.

First Year.—Chemistry—Courses A and B; Chemical Laboratory three days per week; Practical Organic Class during the Summer Term; Technological Chemistry, Course F. Physics, with laboratory work, one day per week. Mathematics (intermediate). German. Engineering, First Year Course, Autumn and Lent Terms. Intermediate B.Sc. Examination may be passed.

Second Year.—Chemistry, Lecture Course G, on Organic Chemistry, Lecture Course E or D, Technological Chemistry, Course F, on Metallurgy. Chemical Laboratory, four days per week. Engineering or Physics (Advanced). The Final Examination for the Victoria B.Sc., or the Intermediate Examination of the Institute of Chemistry, may be taken.

Third Year.—Courses D, F, and C. Any other Courses omitted in a previous year. Laboratory, five days per week. Students may finally choose a special subject either of research or of applied Chemistry. The Final Examination for the Associateship of the Institute of Chemistry of Great Britain and Ireland may be taken. Three years study after passing the Preliminary Examination of Victoria University are required for the B.Sc. Degree in the Honours School of Chemistry.

The Sheridan Muspratt Chemical Scholarship of £50 per annum, tenable for two years, will be competed for in December, 1897, on an Examination in subjects which are included in the first two years of the above curriculum. Other Scholarships, Entrance Scholarships, and Free Studentships are also available to Students.

Evening Classes.

Classes, including laboratory work, will be held on Chemistry, Metallurgy, and on Analysis of Gases.

The Prospectus containing full particulars may be obtained from the Registrar, University College, Liverpool.

DURHAM COLLEGE OF SCIENCE, NEWCASTLE-ON-TYNE.

Professor of Chemistry—P. Phillips Bedson, M.A., D.Sc., F.I.C., F.C.S.

Lecturer in Chemistry—Saville Shaw, M.Sc., F.C.S.

Lecturer in Agricultural Chemistry—R. Greig Smith, M.Sc., F.C.S.

Assistant Lecturer and Demonstrator—F. C. Garrett, M.Sc., F.C.S.

The Session will commence on September 27th, 1897.

1. **General Course.**—This Course of Lectures will extend over the three terms of the Session, and is intended to serve as an introduction to the Science. The Lectures will be of an elementary character, and whilst framed to meet the requirements of First Year Students will also be serviceable to such as intend pursuing Chemistry in its various applications in the arts and manufactures, as, for instance, Brewing, Metallurgy, the Manufacture of Soda, Soap, Glass, &c. The subjects treated will include an exposition of the Principles of Chemistry, and a description of the preparation and properties of the chief Elementary Substances, both metallic and non-metallic, and their more important native and artificial compounds. A section of this Course will be devoted to an outline of Organic Chemistry. The class will meet on Mondays, Wednesdays, and Fridays, at 11 a.m., and will commence on Wednesday, October 6th. Fee, £3 10s. for the Session.

2. **Advanced Course.**—Inorganic Chemistry, Tuesdays 3 to 4 p.m., during the Session. Fee, £2; or for students taking Organic Chemistry, £1.

3. **Organic Chemistry.**—A Course of Lectures will be given throughout the Session, the subject of which will be the Chemistry of the Carbon Compounds and an introduction to Chemical Theory. This class will meet on Tuesdays and Thursdays, at 11 a.m., and Fridays 3 to 4 p.m., and will commence on Thursday, October 7th. Fee, £3 10s. for the Session.

Advanced Classes will be formed for the study of Inorganic, Organic, and Theoretical Chemistry. Fee for the course, £3 10s.

A Lecture Course in Analytical Chemistry will be given on Mondays, at 3 p.m.

Metallurgy and Assaying.—Lecturer, Saville Shaw, M.Sc., F.C.S. A Metallurgical Laboratory is provided, in which instruction is given in the ordinary processes of Dry Assaying, and in the preparation and analysis of Alloys, &c. Fee as for Chemical Laboratory.

Agricultural Chemistry.—The instruction in this branch of Chemistry will consist of a series of Lectures and of special practical work in the Chemical Laboratory. Students will be expected to have a knowledge of Elementary Chemistry, such as may be obtained by attending the General Course.

The Lecture Course in Agricultural Chemistry is

arranged for two days a week throughout the Session. Fee, £3 10s.

Practical Chemistry.—The Laboratory is open from 10 a.m. to 1 p.m., and from 2 to 5 p.m., except on Saturdays, when it closes at 1 p.m. **Laboratory Fees.**—Students working two days, £2 10s. per term, £6 per session; one day per week, £1 10s. per term, £3 10s. per session.

Courses of Study.—Students will be divided into two classes:—(1) Regular, or Matriculated Students, who are also Members of the University of Durham; and (2) Non-Matriculated Students. Regular Students will be required to follow such a course of study in the subjects professed in the College as will enable them to pass the Examinations for the title of Associate in Physical Science of the University of Durham. Non-Matriculated Students will attend such classes as they may select. Every candidate for admission as a matriculated student must pass an examination on entrance, in reading, writing from dictation, English or Latin Grammar, arithmetic (including decimals), and geography. Registered students in medicine are exempted from this examination, or students who produce a certificate of having passed either of the two following examinations:—

1. Durham Examination for certificate of proficiency in General Education, held in March and September.
2. Durham Examination for Students in Arts in their first year, or any examination of a similar nature that may be accepted by the Council.

Associateship in Physical Science.—Every candidate for the Associateship in Physical Science will be required to satisfy the examiners in—Mathematics, Physics, Chemistry, and either Geology or Natural History—in an examination to be held at the end of the candidate's first year. Associates in Science are admissible one year after obtaining the title of Associate to examination for the degree of Bachelor of Science of the University of Durham. **Exhibitions.**—Three Exhibitions of the value of £25, £15, and £10 respectively will be awarded in October next to Candidates desirous of attending the first year course of study in the College.

The examination will be held at the College, and will commence on Wednesday, September 29th.

Evening Lectures.—Courses of Evening Lectures will be given, with a Practical Class for Laboratory instruction.

Two Exhibitions of £15 each will be awarded at the next examination of "Persons not members of the University," which will be held at Durham in March next.

Several other valuable Scholarships are available for students.

OWENS COLLEGE, VICTORIA UNIVERSITY, MANCHESTER.

Professor and Director of the Chemical Laboratory.—Harold B. Dixon, M.A., F.R.S.

Professor of Organic Chemistry.—W. H. Perkin, Ph.D., F.R.S.

Demonstrators and Assistant Lecturers.—George H. Bailey, D.Sc., Ph.D.; Arthur Harden, M.Sc., Ph.D.; P. J. Hartog, B.Sc.; and E. Haworth, M.Sc.

Lecturer in Technical Organic Chemistry.—Jocelyn F. Thorpe, Ph.D.

Assistant Lecturer in Metallurgy.—J. Crowther.

The Session begins on October 5, 1897, and ends on July 2, 1898.

The instruction is given by means of Experimental Lectures and Tutorial Classes. The Chemical Classes form part of the Courses for Chemistry in the University.

Chemistry Lecture Courses.

General Chemistry Course.—Tuesdays, Thursdays, and Saturdays, at 9.30, during the two Winter Terms.

Introduction to Organic Chemistry.—Wednesdays and Fridays, at 9.30, during Lent Term.

These courses are intended for Medical Students and others beginning the study of chemistry.

First Year Honours Course.—Mondays, Wednesdays, and Fridays, 11.30 a.m., during the two Winter Terms. The Non-Metals.

Second Year Honours Course.—Mondays, Wednesdays, Fridays, 3.30 p.m., during the two Winter Terms. The Metals.

Third Year Honours Course.—At times to be arranged. Physical Chemistry.

Organic Chemistry (General).—Mondays and Fridays, 9.30, during two Winter Terms.

Organic Chemistry (Advanced).—Tuesdays and Thursdays, 9.30, during the two Winter Terms.

History of Chemistry and Chemical Philosophy.—Wednesdays, 9.30, during the Session.

METALLURGY.—**Lectures:** The Metallurgy of Copper, Lead, Silver, Gold, and the Metallurgy of Iron and Steel will be given in alternate years. **Practical:** The Laboratory will be open to students every day.

The Chemical Laboratories are open daily from 9.30 a.m. to 4.30 p.m., except on Saturdays, when they are closed at 12.30 p.m.

Courses for B.Sc. Degree.—To qualify for the B.Sc. Degree of the Victoria University, Students have to attend a prescribed course of study extending over three years, and to pass the Preliminary Examination of the University either on entering or at the end of a year's Course.

The Honours Course of Chemistry is as follows:—First year: First year Honours Lectures; Mathematics (3 hours a week); Physics (3 hours a week); a Language (3 hours a week); Chemical Laboratory (3 days per week). Second year: Second year Honours Lectures; General Organic Lectures; Applied Chemistry Lectures; Physics Laboratory (1 day per week); Chemical Laboratory (3 days per week). Third year: Third year Honours Lectures; Honours Organic Lectures; History of Chemistry Lectures; Chemical Laboratory (5 days per week).

The following awards are made to successful Students in the Honours Examination:—A University Scholarship of £50; a Mercer Scholarship of £25. A University Fellowship of £150 is awarded annually among the Graduates in Science for the encouragement of Research. Among the College Scholarships open to Chemical Students are the Dalton Chemical Scholarship, £50 per annum for two years; the 1851 Exhibition Scholarship; the John Buckley Scholarship; &c.

Applied Chemistry.

First Course.—Sulphuric Acid and Alkali Manufactures. General Principles of Chemical Engineering.

Second Course.—The Chemistry of Fuel. The Manufacture of Illuminating Gas and Gaseous Fuel.

Third Course.—Natural and Artificial Dye-stuffs, and the Principles of Dyeing and Printing.

Certificates in Applied Chemistry.

The course extends over a period of three years, and comprises systematic instruction by means of lectures and practical work in the laboratories.

Before admission to the first year's course students are required to give such evidence of elementary knowledge of Mathematics and Chemistry as shall be considered satisfactory by the Senate.

The first year's course is the same for all students working for the certificate.

In the second and third years a choice may be made between Inorganic and Organic Chemistry. By this division of the subject a student wishing to apply himself specially to the inorganic side of the science, may attend during his second year the Honours course in Metals, and courses on Geology or Mineralogy, and during his third year, courses on Metallurgy and on Geology or Mineralogy; while a student wishing to apply himself specially to the organic side of the science, may attend during his second and third years the Courses on Organic Chemistry, and courses on the Coal Tar Colours and on Dyeing and Printing.

Part of the Laboratory practice in the second and third years will consist in the examination and analysis of raw materials, products from chemical works, &c., in connection

with the special courses of lectures on Applied Chemistry. In the Chemistry and Physical laboratories the practical work in the second year will be arranged in accordance with the branch of Chemistry selected by the candidate.

In the third year the student, if sufficiently advanced, will be set to work on some analytical process or problem in Applied Chemistry, under the direction of the teaching staff.

UNIVERSITY COLLEGE, NOTTINGHAM.

DEPARTMENTS OF CHEMISTRY AND METALLURGY.

Professor of Chemistry—Frank Clowes, D.Sc. Lond., F.I.C.

Demonstrators of Chemistry—J. J. Sudborough, D.Sc., Ph.D., F.I.C.; R. M. Caven, B.Sc., F.I.C.; and G. Meland, B.Sc., A.R.S.M., F.I.C.

The Classes of the College are open to students of both sexes above sixteen years of age.

The Session commences on October 11th.

Lecture Courses.—The Chemistry Day Lectures extend over three years. In the first year a student enters for the course on Non-Metals for the first two terms and for Elementary Organic Chemistry in the third term. In his second year he takes the course on Metals for the first two terms. In his third year he attends a course on Advanced Organic Chemistry or Applied Chemistry. Fee for Day Lectures and Classes: Non-Metals or Metals 42s.; Organic Chemistry (one term) 21s.; Advanced Organic Chemistry, 21s. per term.

Demonstrations and Lectures on Analytical Chemistry will be given in the day and evening, and should be attended by all students.

A Chemical Calculation Class is also held. Fee per Term, 5s.

Students may qualify themselves by attendance at these lectures and classes for the Examinations of the Universities of London, Cambridge, or Oxford, and for the Medical Examinations of the Royal College of Surgeons and of the Universities of Cambridge and Edinburgh: they may also obtain instruction in Chemistry for technical or other purposes, and can enter for a full Chemical Engineering Curriculum. Special attention is given to the requirements of candidates for the Associateship of the Institute of Chemistry.

Practical Chemistry and Metallurgy.—The Chemical and Metallurgical laboratories are open every day from 9 to 5, except on Saturday, when the hours are from 9 to 1; also on Tuesday and Thursday evenings from 7 to 9. Each Student works independently of other Students at a course recommended by the Professor. Instruction is given in general Chemical Manipulation, in Qualitative and Quantitative Analysis, and in the methods of Original Chemical Investigation and Research; Students are also enabled to work out the applications of Chemistry to Pharmacy, Metallurgy, Dyeing, Agriculture, Brewing, Iron and Steel, Tanning, and other Manufacturing Processes. Fees for day students: For one term, £7; for the session, £18; for six hours weekly 40s., and 5s. extra for each additional hour per week. For evening students, 10s. for two hours per week, three hours 15s., four hours 20s., six hours 30s., per term.

Courses of Technical Chemistry Lectures are also given on Engineering, Dyeing and Bleaching, Brewing, Plumbing, Bread-making, Gas Manufacture, and on other processes of applied Chemistry.

Pharmaceutical Students can at all times work in the Chemical Laboratory, taking work suitable for the preparation for the Minor Examinations. Special lectures will also be given in Chemistry and Materia Medica.

Government Lectures and Classes.—Evening Lectures and Laboratory instruction will be given by the Demonstrators of Chemistry to Students who intend to present themselves for Examination by the Government Science and Art Department in May next. Inorganic, organic, and practical chemistry, agricultural chemistry, and metallurgy will be taught in the elementary, advanced, and

honours stages, each of which commences at the beginning of the College Session in September. Fee for each Lecture Course, 5s.; for each Laboratory Course, 10s.

An *Agricultural Course* of instruction, extending over two years, is now organised under the general direction of Mr. M. J. R. Dunstan, M.A., F.R.S.E. It includes instruction in chemistry, botany, agriculture, with practical work on experimental fields, dairy work, farriery, land surveying, &c. The instruction is designed for those who intend to become farmers, bailiffs, land agents, or colonists, and may be extended to a third year if desired. Fee, £15 per annum for residents in Notts, £20 to residents in other counties.

Full information concerning all College Classes is given in the College Prospectus, price one penny.

UNIVERSITY COLLEGE, SHEFFIELD.

Professor of Chemistry—W. Carleton Williams, B.Sc., F.C.S.

Demonstrators and Assistant Lecturers—G. Young, Ph.D., and L. T. O'Shea, B.Sc., F.C.S.,

The Session will commence on October 6th.

First Course.—Chemistry of the Non-Metallic Elements. Tuesday and Friday from 10 to 11 a.m. Fee, £2 12s. 6d.

Second Course.—Chemistry of Metals. Monday and Thursday from 10 to 11 a.m. £2 12s. 6d.

Third Course.—Inorganic Chemistry, Honours Course, Monday, 3. Fee, £1 11s. 6d.

Organic Chemistry.—Elementary: Wednesdays, 10 to 11; fee, 15s. Honours: Tuesdays and Thursdays, 12 to 1; fee, £2 12s. 6d. Advanced: Thursdays, 4 to 5; fee, £1 11s. 6d. Special Course: Hours to be arranged; £1 11s. Chemistry of the Colouring Matters: Fridays, 12 to 1; fee, £1 11s. 6d.

Short Courses of Lectures are also given by L. T. O'Shea on the Chemistry of Coal Mining, and on Thermic Chemistry.

A Course of Lectures is arranged for Medical Students, with a special class in Qualitative Analysis.

Laboratory.—Working hours to be arranged between Professor and Students.

Sessional Fees for Day Students:—Six hours per week, £5 5s.; Nine, £7; Twelve, £8 8s.; Eighteen, £11 5s.; Twenty-four, £14; Thirty-two, £17.

Day Students may not enter for less than six hours a week. Students joining the Laboratory at Christmas will be charged two-thirds and at Easter one-third of the Fees for the whole Session.

Fees for short periods (working thirty-two hours per week):—For one month, £3 3s.; two months, £5 5s.

An arrangement has been entered into with the Science and Art Department, South Kensington, which will enable Science Teachers to work in the Chemical Laboratory for three, six, or twelve hours a week on payment of one-quarter of the usual fee, the Department being willing to pay the remainder under certain conditions, of which full information may be obtained on application to the Registrar.

Evening Classes.—Lectures, Wednesday, 8 to 9. Laboratory instruction, Wednesday, 6 to 9, and another series to be arranged if desired. Sessional Fee, one evening per week, £1 10s.; two, 50s.; or Lecture Class and Laboratory, on Wednesday evening, £1 10s. Fee for one term, 17s. 6d.

UNIVERSITY COLLEGE, DUNDEE.

UNIVERSITY OF ST. ANDREWS.

Professor of Chemistry—James Walker, Ph.D., D.Sc.

Assistant Lecturer—J. S. Lumsden, Ph.D., B.Sc.

Lecture Assistant and Laboratory Steward—J. Foggie, F.C.S.

The Winter Session begins on October 6th, and ends on March 16th. The Summer Session extends from the middle of April to the end of June.

The *First Year's Lecture Course on Systematic Chemistry* is given daily during the Winter Session, and

embraces the Elements of Inorganic and of Organic Chemistry.

Advanced Courses, of about fifty lectures each, will be given during the year as follows:—

Organic Chemistry; Inorganic Chemistry, including the more important technological applications; Theoretical and Physical Chemistry; Bleaching and Dyeing, including the Chemistry of the Textile Fibres.

Practical Instruction in all of the above branches will be given in the Laboratories and Dye-house. Special facilities are afforded to Research Students.

The Lectures and Laboratory Practice in Chemistry are recognised by the Medical Colleges of London and Edinburgh. The Courses are suitable for the degrees of the University of London and for the Civil Service appointments, and will also satisfy the requirements of Students in Pharmacy, and of Students who intend to become candidates for the Associateship of the Institute of Chemistry, as far as qualification in Chemistry is concerned.

UNIVERSITY OF EDINBURGH.

DEPARTMENT OF CHEMISTRY.

Professor—Alex. Crum Brown, M.D., D.Sc., F.R.S.

Lecturers—L. Dobbin, Ph.D., and H. Marshall, D.Sc.

Assistants—W. W. Taylor, M.A., B.Sc., and J. P. Longstaff.

The working terms are—Winter Session, from middle of October to middle of March; Summer Session, from beginning of May to end of July.

Lecture Courses.—During the Winter Session a General Course of Chemistry for medical and science students is given by the Professor. The class meets daily; fee £4 4s. An Advanced Course of twenty-five lectures is also given in the Winter Session; fee, £2 2s. A class on Organic Chemistry is held in summer; fee, £2 2s. There is also a class on Chemical Theory, by Dr. Dobbin; fee £1 rs.; and a class on Mineralogy and Crystallography, by Dr. Marshall; fee, £2 2s. All these Lectures, except the General Course, are now open to women.

In addition to the above, Lecture Courses are given by the Assistants on some particular branch of Organic and Inorganic Chemistry. These Lectures are free to Laboratory Students.

Tutorial classes are held in connection with the General Course.

Laboratories.—Practical classes for Medical Students meet daily during the latter part of the Winter Session and in the Summer Session. (Fee, £3 3s.) The laboratories for analytical and advanced practical work are open daily from 9.30 till 4.30. (Fees: Whole Day—Winter Session, £10 10s., Oct.-Dec., Jan.-March; or Summer Session, £5 5s. Half Day—Winter Session, £6 6s., Oct.-Dec., Jan.-March; or Summer Session, £3 3s. Preference will be given to students in the above order. Students who are not Matriculated may attend the Chemical Laboratory on payment of the entrance fee of 5s. in addition to the Laboratory fees. Full Courses of instruction are given in Analytical, Practical Organic and Inorganic Chemistry, including Gas Analysis, Metallurgy, and Assaying. Facilities are afforded to advanced students who desire to undertake chemical investigations.

Various prizes and scholarships are attached to the laboratory and general class.

Graduation.—Two Degrees in Pure Science are conferred, viz., Bachelor of Science (B.Sc.) and Doctor of Science (D.Sc.).

Candidates for Degrees in Science, if not graduates (by examination) in Arts in one of the Universities of the United Kingdom or in a Colonial or Foreign University recognised for the purpose by the University Court, must pass a preliminary examination in (1) English; (2) Latin, Greek, French, or German; (3) Mathematics; (4) One of the languages Latin, Greek, French, German, Italian, not already taken under (2), or Dynamics. In the case of a student whose native language is other than European, the Senatus may, at the Preliminary Examination, accept

such language as a substitute for a modern European language. The Senatus may also in such a case accept as an alternative to Latin or Greek any other classical languages, such as Sanscrit or Arabic.

The First B.Sc. Examination embraces Mathematics, or Biology (i.e., Zoology and Botany), Natural Philosophy, and Chemistry. The Second B.Sc. Examination includes any three or more of the following subjects:—Mathematics, Natural Philosophy, Astronomy, Chemistry, Human Anatomy, including Anthropology, Physiology, Geology, including Mineralogy, Zoology, including Comparative Anatomy, and Botany, including Vegetable Physiology. Chemistry in this examination embraces Inorganic, including Mineralogical, Chemistry; Organic Chemistry; Physical Chemistry; Chemical Crystallography; History of Chemistry. Practical Examination:—Complex Qualitative Analysis; Quantitative Analysis, including Gas Analysis and Organic Analysis; Preparation of Pure Substances, organic and inorganic; Physico-chemical Measurements.

In the Courses for Final Examination in Pure Science, two written papers are set in each subject professed, the second of a higher standard than the first. Candidates must pass the first section in all, and the second section in at least one, of the subjects professed; the same regulations apply also to the Practical and Oral Examinations.

A candidate for the D.Sc. Degree must submit a thesis on original work done by him. The Thesis must be approved before the candidate is allowed to proceed to Examination. The candidate in Chemistry may be required to pass a searching examination in one of the following branches:—(1) The Chemistry and Chemical Technology of Inorganic Bodies, including Metallurgy; (2) Organic Chemistry; and to show a thorough practical acquaintance with chemical analysis in all its branches, and with the preparation of pure substances.

HERIOT-WATT COLLEGE, EDINBURGH.

Professor—John Gibson, Ph.D., F.R.S.E.

Assistant Professor—(Vacant).

Demonstrators—Andrew F. King and James B. Shand.

The Session begins October 5th, 1897.

The curriculum of this College comprises both Day and Evening Classes, each department providing the higher general and technical education.

Chemistry.—The first course for day students is a combination of Lectures with Laboratory instruction. In the Lectures some of the more important elements and their compounds are discussed in detail, so as to lead to a knowledge of the general laws of chemical action. Other important elements are treated in less detail, and the relations and classification of the elements generally are broadly indicated. In the Laboratory each student will receive instruction in general chemical manipulation, in accurate weighing, volumetric measurements, and in some of the simpler methods of quantitative analysis. After making a series of simple preparations, he works through a number of experimental exercises illustrating chemical combination, oxidation, reduction, and double decomposition. These exercises are followed by instruction in simple methods of qualitative analysis, especial attention being given to dry way testing and the use of the spectroscope. Students attending a further course may take up the study of systematic analysis, and extend the knowledge they have gained of quantitative analysis by exercises in gravimetric, volumetric, and electrolytic methods. Ultimately they may make a speciality of any branch of the subject which may be most necessary for their future work. Great attention has been paid to the thorough equipment of Advanced Laboratories, and special facilities are given to advanced students who may wish to engage in any class of Research (Inorganic or Organic) whether of a purely chemical or of a technical nature.

The teaching in the Evening Classes is based on the Syllabus of the Science and Art Department, and in-

cludes Elementary, Advanced, and Honours Courses in Theoretical and Practical Inorganic and Organic Chemistry.

GLASGOW AND WEST OF SCOTLAND TECHNICAL COLLEGE.

Professor of Chemistry—G. G. Henderson, D.Sc., M.A.
Professor of Technical Chemistry—E. J. Mills, D.Sc., F.R.S.

Agricultural Chemistry Lecturer—John W. Paterson, B.Sc., Ph.D.

Professor of Metallurgy—A. Humboldt Sexton, F.C.S., F.R.S.E.

The main objects of this College are to afford a suitable education to those who wish to qualify themselves for following an industrial profession or trade, and to train teachers for technical schools. It was founded by an Order in Council, dated 26th November, 1886, according to a scheme framed by the Commissioners appointed under the provisions of the Educational Endowments (Scotland) Act, whereby Anderson's College, the Young Chair of Technical Chemistry in connection with Anderson's College, the College of Science and Arts, Allan's Glen's Institution, and the Atkinson Institution were placed under the management of one governing body.

The Diploma of the College is awarded to Day Students who have attended prescribed courses of instruction and passed the necessary examinations. The ordinary courses extend over three years, but arrangements are made for advanced students continuing their studies in special departments.

Complete courses of instruction in Metallurgy and Mining will be given in both Day and Evening Classes.

Copies of the Calendar for 1896-97 may be had from Mr. John Young, B.Sc., the Secretary, 38, Bath Street, Glasgow, price by post, 1s. 4d.

UNIVERSITY OF ST. ANDREWS.

UNITED COLLEGE OF ST. LEONARD AND ST. SALVATOR.

Professor of Chemistry—T. Purdie, B.Sc., Ph.D., F.R.S.

The Session begins on October 6th. A Competitive Examination, open to intending Students of Arts or Science, for about fifty Entrance Bursaries, ranging in value from £40 to £10 each per annum, will be held on September 25th and following days. About thirty of these Bursaries are restricted to Men and twenty to Women, the latter being intended for women who at the conclusion of their Arts or Science Course will proceed to Medicine. Two are open to students of either sex. Two Scholarships of £100 each, tenable for one year, will be open for competition to Graduates of Science at the close of Session 1897-98. A Hall of Residence is provided for Women Students. Two Degrees in Science are conferred by the University of St. Andrews, viz., Bachelor of Science (B.Sc.) and Doctor of Science (D.Sc.), and Chemistry is also included in the curriculum for the M.A. Degree; the regulations will be found in the "University Calendar."

Lecture Courses.

Two distinct Courses of Lectures are given, each comprising at least one hundred meetings of the class.

First Year's Course.—This Class meets at 11 o'clock on five days in the week. The introductory lectures treat of the Nature of Chemical Action, the Classification of Substances into Elements and Compounds, the Phenomena of Oxidation, and the Composition of Air and Water. The Laws of Chemical Combination and the Atomic Theory are next discussed, after which the more commonly occurring elements and inorganic compounds are described systematically. Elementary Organic Chemistry is also included in the Course.

The chemistry of manufactures is referred to only cursorily; special attention, on the other hand, is

given to those parts of the science which are of general educational value, and as much of the theory of chemistry is introduced as is compatible with elementary treatment. The Lectures are supplemented by a short Course of Laboratory Practice, intended to illustrate the principles of the science.

These courses of instruction are intended to meet the requirements of the Arts' Curriculum; also of candidates for the First B.Sc. Examination, and of students of medicine, so far as Theoretical Chemistry is concerned.

Second Year's Course.—The first part of the Course is devoted to Organic Chemistry, and the second part treats of the General Principles and Theory of Chemistry, and of more advanced Inorganic Chemistry, the instruction in general being such as is required for the Second B.Sc. Examination.

Certificates are awarded on the results of examinations, and the "Forrester Prize" of about £10 is awarded to the best Student of the year.

Fee for the Session, for each Course, £3 3s.

Practical Chemistry.

The Laboratory is open daily from 9 a.m. to 4 p.m., except on Saturdays, when it is closed at 1 p.m. The work pursued in the Laboratory comprises:—

(1) The performance of experiments illustrative of the Principles of Inorganic and Organic Chemistry; (2) Qualitative and Quantitative Analysis; (3) Original Investigation. Each student pursues an independent course of study under the supervision of the Professor or Demonstrator, the nature of the work varying with the proficiency of the student and the particular object he may have in view. Suitable courses of instruction in Practical Chemistry are provided for candidates for the First and Second B.Sc. Examinations, and for Students of Medicine.

The fees for Practical Chemistry vary according to the number of hours taken weekly. A certain number of working places in the Laboratory will be available without fee for students who are capable of undertaking original investigation.

QUEEN'S COLLEGE, BELFAST.

Professor—E. A. Letts, Ph.D., D.Sc., F.R.S.E., &c.

The Session commences on Tuesday, October 19, 1897.

I.—Chemistry.—The lectures are delivered at 3 p.m., on the first five days of each week until the beginning of April, and on three days of each week after May 1st, at 2 p.m. The course is divided into three parts:—(1) Chemical Philosophy; (2) Inorganic Chemistry; (3) Organic Chemistry. Fee, £2.

II.—Practical Chemistry.—In this course the Students are instructed in the general methods of conducting Chemical Analyses. Fee, £3.

III.—Laboratory Pupils.—The Chemical Laboratory is open from November until the end of March, and from May 1st until the third week of July, on the first five days of the week, from 10 a.m. until 4 p.m. Students are admitted as working pupils on payment of a fee of £5 for the first period, or of £3 10s. for the second period (or for a single term).

Scholarships.—In addition to various Scholarships awarded in the Faculties of Arts and Medicine in which Chemistry forms a part of the examination, there are other valuable Scholarships awarded specially in connection with the schools of Chemistry and Physics.

QUEEN'S COLLEGE, CORK.

Professor—Augustus Edward Dixon, M.D.

Demonstrator—R. E. Doran, F.C.S.

The College Session begins on October 19th, 1897, and ends on June 11th, 1898. The classes are open to male and female students.

Systematic Chemistry.—(1) General course of Inorganic Chemistry, Elementary Organic Chemistry, and Chemical Philosophy.—Fee for each Sessional Course, £2. Each

subsequent Course, £1. (2) Advanced Organic Chemistry, and Chemical Philosophy.

Practical Chemistry.—(1) Two ordinary Courses of Practical Chemistry will be held, each of three months' duration; one commencing on January 3rd, 1898, and adapted to the requirements of Students proceeding to the Examinations of the Royal University of Ireland; the other ending about the last week in June, and suitable for Medical Students intending to present themselves for the Examinations of other Licensing Bodies. Fee for each Sessional Course, £3. (2) A Course for Pharmaceutical Students will be held in the second and third terms; fee, £5. (3) Special Courses.

The Chemical Laboratory is open daily from 10 to 4 o'clock (except during class hours and on Saturdays) under the Superintendence of the Professor, to Students entering for special courses of qualitative and quantitative analysis; organic chemistry; or for the purpose of original investigation.

QUEEN'S COLLEGE, GALWAY.

Professor—Alfred Senier, Ph.D., M.D., F.I.C.

Demonstrator—Hugh Ryan, B.A.

The College Session is divided into three terms. The First Term extends from October 19 to December 22, the Second Term from January 6 to April 2, and the Third Term from April 18 to June 11.

Chemistry is studied by attendance at Lectures, by work in the Laboratories, and by the use of the College Library. The Courses in the several faculties are arranged with a view to the requirements of the Royal University of Ireland, but are adapted also to those of other Universities and licensing bodies.

Lecture Courses. Faculty of Arts.—1. Second year's Course, Inorganic and the Elements of General Chemistry. 2. Third year's Course, Advanced Organic Chemistry. 3. Fourth year's Post-Graduate Course, Advanced General Inorganic and Organic Chemistry. **Faculty of Medicine.**—First year's Course, Inorganic and Elementary Organic Chemistry. **School of Engineering.**—First year's Course, Inorganic Chemistry.

Laboratory Courses. Faculty of Arts.—1. Second year's Course, Exercises in Inorganic Qualitative Analysis. 2. Third year's Course, Quantitative Analysis and other experiments to suit the requirements of individual Students. 3. Fourth year's Post-Graduate Course, Advanced Quantitative Analysis, Organic and Inorganic Preparations, and determination of their Physical and Chemical characters. 4. The Laboratories are also open to Students for work in other branches of Chemistry. **Faculty of Medicine.**—1. Second year's Course, Inorganic and Organic Elementary Qualitative Analysis, and the Chemical Examination of Urine. **School of Engineering.**—1. Second year's Course, Inorganic Qualitative Analysis.

For Fees, Regulations as to Scholarships, and other particulars apply to the Registrar, from whom the Calendar, published in December, and the Extracts from Calendar, published in advance in July, may be obtained.

ROYAL COLLEGE OF SCIENCE FOR IRELAND, STEPHEN'S GREEN, DUBLIN. (SCIENCE AND ART DEPARTMENT).

Professor of Chemistry—W. N. Hartley, F.R.S.

Assistant Chemist—Hugh Ramage, F.I.C., Associate of the Royal College of Science, Dublin.

Demonstrator of Chemistry and Assaying—J. Holms Pollok, B.Sc.

The Session commences on Tuesday, October 5th, 1897. The Royal College of Science for Ireland supplies, as far as practicable, a complete course of instruction in Science applicable to the Industrial Arts, and is intended also to aid in the instruction of teachers for the local Schools of Science.

Diplomas are awarded in the Faculties of Mining, Engineering, and Manufactures, Physics, and Natural Science. The Diploma of Associate of the Royal College

of Science in the Faculty of Manufactures is recognised by the Council of the Institute of Chemistry of Great Britain and Ireland as qualifying candidates for admission to the practical examinations of the Institute.

The instruction in Chemical Science includes (1) General Chemistry; (2) Advanced Chemistry, including Chemical Manufactures and Metallurgy; (3) Analytical and Experimental Chemistry; (4) Instructions in Chemical Research.

Fees payable by Non-Associates:—£2 for each separate Course of Lectures. For Analytical Chemistry and Research—£2 for a special course of one month; £5 for three months; £9 for six months; £12 for the entire session. For Assaying—£5 for three months; £9 for six months; £12 for the entire session.

NOTE.—Important changes have been made in the Curriculum for Associate Students. Full particulars are contained in the Directory of the College, which may be had on application to the Secretary.

The following are supplementary courses of instruction arranged for those who are attending a Course of Lectures:—

- (1) Laboratory Instruction in the Theory of Chemistry.
- (2) An Analytical Course for Students in Engineering.
- (3) A Course of Practical Chemistry for Medical Students.
- (4) The Analysis of Water, Air, Food, and Drugs, intended for the instruction of Public Analysts and Medical Officers of Health.
- (5) Assaying.

There are four Royal Scholarships of the value of £50 each yearly, with Free Education, including Laboratory Instruction, tenable for two years; two become vacant each year; they are awarded on the results of their examinations to Associate Students, not being Royal Exhibitioners, who have been a year in the College. There are also nine Royal Exhibitions attached to the College, of the yearly value of £50 each, with Free Education, including Laboratory Instruction, tenable for three years; three become vacant each year, and are competed for at the May Examinations of the Department of Science and Art.

CHEMICAL LECTURES, CLASSES, AND LABORATORY INSTRUCTION.

CITY AND GUILDS OF LONDON INSTITUTE FOR THE ADVANCEMENT OF TECHNICAL EDUCATION.—The operations of the City and Guilds of London Institute are divided broadly into four branches: the educational work of three London Colleges, and of the Technological Examinations. Programmes of the London Colleges may be had on application to the Head Office of the Institute, Gresham College, Basinghall Street, London, E.C., or from the respective Colleges. The Technological Examinations (Examinations Department, Exhibition Road, S.W.), are conducted once every year at various centres throughout the kingdom. Programme, with Syllabus of Subjects, &c., may be obtained of Messrs. Whittaker and Co., Paternoster Square, London, or through any bookseller, price 10d., net. — *City and Guilds Technical College, Exhibition Road.*—Professor of Chemistry, H. E. Armstrong, Ph.D., F.R.S. The object of this Institution is to give to London a College for the higher technical education, in which advanced instruction shall be provided in those kinds of knowledge which bear upon the different branches of industry, whether Manufactures or Arts. The main purpose of the instruction given is to practically demonstrate the application of different branches of science to various manufacturing industries. In order that this instruction may be efficiently carried out, the Institution, in addition to the lecture theatres and class rooms, is fitted with laboratories, drawing offices, and workshops; and opportunities are afforded for the prosecution of original research, with the object of the more thorough training of the students, and for the elucidation of the theory of industrial processes. The

courses of instruction are arranged to suit the requirements of—1. Persons who are training to become Technical Teachers; 2. Persons who are preparing to enter Engineers' or Architects' offices, or Manufacturing works; 3. Persons who desire to acquaint themselves with the scientific principles underlying the particular branch of industry in which they are engaged. The Matriculation Examinations will begin on Tuesday, Sept. 21st, and the Winter Session opens on Monday, October 4th. *City and Guilds Technical College, Finsbury.*—Professor of Chemistry, Raphael Meldola, F.R.S. The operations of the Technical College, Finsbury, are divided into two distinct portions: Day Classes for those who are able to devote one, two, or three years to systematic technical education; Evening Classes for those who are engaged in industrial or commercial occupations in the daytime and who desire to receive supplementary instruction in the application of Science and of Art to the trades and manufactures in which they are concerned or employed. Each Professor is assisted by Demonstrators. Besides these there are Lecturers and Teachers in special subjects. An examination for the admission of Students will be held at the College at 10 o'clock on Tuesday, September 21st, 1897. *South London Technical Art School.*—Classes in Modelling, Drawing and Painting from the Life, and House Decoration.

CITY OF LONDON COLLEGE, White Street, Moorfields.—Courses of Evening Lectures and Laboratory Practice in Chemistry and Physics, conducted by Mr. I. S. Scarf, F.I.C., F.C.S., assisted by Messrs. H. W. Harris, F.C.S., H. V. Buttfield, F.C.S., and C. A. West, A.R.C.S., F.C.S. Session commences September 27.

BATTERSEA POLYTECHNIC.—Principal, Mr. Sidney H. Wells. Wh. Sc. Inorganic, Organic, and Technological Chemistry, Mr. W. A. Bone, D.Sc. (Vit.), Ph.D., assisted by Mr. J. Wilson, M.Sc. (Vit.). Day and Evening Classes in Science and Art subjects.

BIRKBECK LITERARY AND SCIENTIFIC INSTITUTION, BREM'S BUILDINGS, CHANCERY LANE.—Chemistry Courses will be conducted, commencing September 27, adapted for the Elementary, Advanced, and Honours Examinations of the Science and Art Department, and for the Matriculation, B.Sc., and M.B. Degrees of the London University, by Mr. J. E. Mackenzie, Ph.D., B.Sc.

IMPERIAL COLLEGE OF CHEMISTRY AND PHARMACY, 51, Imperial Buildings, Ludgate Circus.—Mr. F. Davis, B.Sc. *BOROUGH POLYTECHNIC INSTITUTE, St. George's Circus.*—Mr. F. Mollwo Perkin, Ph.D. Lectures and Laboratory work in Chemistry and Physics. Session commences Monday, September 27, 1897.

BRITTON SCHOOL OF CHEMISTRY AND PHARMACY, 12, Knowle Road, Brixton.—Dr. A. B. Griffiths, F.R.S.E., F.C.S., &c.

METROPOLITAN COLLEGE OF PHARMACY, 162, Kennington Park Road, S.E.—Principal, W. Watson Will, F.C.S.

SOUTH-WEST LONDON POLYTECHNIC, Manresa Road, Chelsea.—Principal, Herbert Tomlinson, B.A., F.R.S. Technical Day Classes in Chemical Industries, commencing September 28th.

THE GOLDSMITHS' INSTITUTE, New Cross, S.E.—Head of the Chemistry Department, Mr. W. J. Pope; Assistants, Mr. S. J. Peachy and others. Lectures and Practical Classes in General Chemistry, also in Chemistry applied to Gas Manufacture and other industries, are held in the evenings from 7.30 to 10.0, and are open to both sexes. Special attention is paid to Technical Laboratory work and the investigation of manufacturing difficulties.

UNIVERSITY TUTORIAL COLLEGE, 32, Red Lion Square, Holborn, W.C. (Science Department of the Univ. Coll.).—Chemical, Biological, and Physical laboratories, Morning, Afternoon, and Evening Classes. Students may work either for long or short periods, either for examination or for private practice. The Laboratories accommodate over 100 Students.

SOUTH LONDON SCHOOL OF PHARMACY, Lim., 325, Kennington Road, S.E.—Lectures on Chemistry and Physics, by Dr. John Muter, F.R.S.E., F.I.C., and Mr. J. Thomas, B.Sc. (Lond.), Daily, at 12 noon. Lectures on Botany daily at 1 p.m. and at 2.30 p.m. on Materia Medica and Pharmacy, by Mr. Dodd, F.C.S. The Laboratories for Qualitative and Quantitative Analysis open daily from 9 till 5, under the direction of Mr. de Koningh, F.I.C., F.C.S. The Students' Laboratory of this Institution is specially designed to accommodate 40 Students. The Technical Laboratory is open daily from 9 till 5, and is fully fitted with all apparatus for teaching the manufacture of drugs and chemicals. Periodical Examinations of the Students are held by Visiting Examiners appointed by the Council of Education, and Medals and Certificates are awarded on the results thereof. Fees for the first three months 12 guineas; afterwards 3 guineas per month, inclusive of all departments.

EAST LONDON TECHNICAL COLLEGE, People's Palace, E.—Chemistry: Professor, J. T. Hewitt, M.A., D.Sc., Ph.D.; Demonstrator, F. G. Pope; Assistants, H. A. Phillips and W. T. Gidden. Lectures and Practical Classes are held in the daytime in connection with the three years' course of the Day Technical College. Evening Classes are also held, offering instruction in the courses of the Science and Art Department and for the examinations of the University of London.

POLYTECHNIC INSTITUTE, 309, Regent Street, London, W.—Mr. R. A. Ward and Assistants.—Evening Classes in Theoretical and Practical Chemistry, &c. The Classes are open to both sexes. The next term commences on Monday, September 27th.

WESTMINSTER COLLEGE OF CHEMISTRY AND PHARMACY, Trinity Square, Borough, S.E.—Messrs. Wills and Wootton. Day and Evening Classes.

THE CLIFTON LABORATORY, Berkeley Square, Bristol.—Principal, E. H. Cook, D.Sc. (Lond.), F.I.C. Students are received either as Private Pupils or Members of a Class. Instruction is given to those requiring to use science or scientific methods in Commercial and Industrial pursuits, or in preparing for Examinations. Students are urged to undertake researches and receive special attention from the teachers. Every effort is made to produce thorough chemists rather than successful examinees.

LEEDS TECHNICAL SCHOOL (late School of Science and Technology), Cookridge Street.—Head Master and Lecturer on Chemistry, Mr. R. E. Barnett, B.Sc., A.R.C.S., assisted by Mr. R. W. Ferguson, A.R.C.S. Evening Courses are offered in Inorganic and Organic Chemistry, both theoretical and practical. The Laboratory is well equipped, and arrangements are made for pharmaceutical study, research, or other special work, for which purpose it is open also in the daytime. In Metallurgy, Lecturer Mr. B. A. Burrell, F.I.C., F.C.S., elementary, advanced, and honours Courses of Lectures and Laboratory work are held. In Physics, Lecturer Mr. J. E. Tindall, B.Sc., theoretical and practical classes are held, and in the latter provision is made for candidates for Inter. Sci. and B.Sc. There are also Courses in Engineering, Botany, Geology, and several Technological subjects. The new Session commences at 7.0 p.m. on Monday, September 20th. Fee for any Evening Course of Lectures:—Elementary, 2s. 6d.; Advanced or Honours, 3s. 6d.; Laboratory Courses from 5s. to 15s.

THE MUNICIPAL TECHNICAL SCHOOL, Princess Street, Manchester.—Theoretical and Practical Chemistry, Mr. E. Knecht, Ph.D., F.I.C., Mr. J. Grant, F.I.C., F.C.S., Mr. L. G. Radcliffe, and Mr. J. Allan. Metallurgy, Mr. E. L. Rhead. At this important Municipal School, with an attendance of upwards of 3500 Students, there are organised Day Courses in Pure Chemistry, with applications to Dyeing, Bleaching, Printing, Brewing, and Metallurgy. In addition there are Evening Courses, not only in Pure Chemistry, but in Metallurgy, Mineralogy, Iron and Steel Manufacture, Brewing, Bleaching, Dyeing,

and Printing, Coal Tar Products, Paper Manufacture, Gas Analysis, Gas Manufacture, Chemical Engineering, Chemistry for Engineers and Builders, and Photography. The complete Syllabus (4d., by post 6d.) may be obtained on application to Mr. J. H. Reynolds, Director and Secretary, Princess Street, Manchester.

THE MANCHESTER COLLEGE OF PHARMACY, Oxford Street, Manchester.—Director, Mr. Charles Turner, F.C.S. Classes and Lectures specially designed to meet the requirements of Pharmaceutical Students.

CHEMICAL AND BACTERIOLOGICAL LABORATORY, Barton Arcade, Manchester.—Mr. George J. Allen, F.C.S.—Special Instruction and Practice in Applied Science.

HIGHER GRADE SCHOOL, PATRICROFT.—Science and Art Day and Evening School, and Institute for Women. Demonstrator in Chemistry, Mr. R. J. B. Sanderson.

SHEFFIELD PHARMACEUTICAL AND CHEMICAL SOCIETY, New Surrey St.—Chemistry and Physics, Mr. H. Highfield. Materia Medica and Botany, Mr. John Austen and Mr. E. C. Exell. Session commences first week in October.

HARTLEY COLLEGE, SOUTHAMPTON.—Principal, R. Wallace Stewart, D.Sc. (Lond.). Lecturer in Chemistry, D. R. Boyd, B.Sc., Ph.D. Session commences Tuesday, September 28, 1897.

STOCKPORT TECHNICAL SCHOOL.—Department of Chemistry and Dyeing.—Principal: Mr. R. J. Brown, M.Sc. A syllabus with full particulars of the courses of instruction, hours, fees, &c., is obtainable on application. Special instruction is given in the Dyeing of Felt, &c., as applied to Hat Manufacture.

TECHNICAL INSTITUTE, SWANSEA.—Classes in Theoretical and Practical Organic and Inorganic Chemistry, Metallurgy, Hygiene, Mathematics, Physiology, Sound, Light, and Heat, from October to May. Principals, D. J. Morgan, B.A. (Cantab.), C. A. Seyler, B.Sc., F.I.C.

ABERDEEN UNIVERSITY.—Prof. Japp. **SCHOOL OF MEDICINE, Edinburgh**.—Dr. S. Macadam, Mr. King, Mr. I. Macadam, and Drs. Aitken and Readman. **ST. MUNGO'S COLLEGE AND SCHOOL OF MEDICINE, EDINBURGH**.—Dr. Marshall.

SURGEON'S HALL, Nicolson Street, Edinburgh.—Dr. Stevenson Macadam, Lecturer. Classes for Medical and General Students. Lectures commence Tuesday, October 12, 1897.

GLASGOW UNIVERSITY.—Prof. J. Ferguson. **ANDERSON'S COLLEGE, GLASGOW**.—Mr. J. R. Watson.

ROYAL COLLEGE OF SURGEONS IN IRELAND, DUBLIN.—Professor of Chemistry and Hygiene: Sir Charles A. Cameron, M.D., F.R.C.S.I. Instruction is given in the College Laboratory in General, Practical, and Analytical Chemistry, and in the subjects (Physical, Chemical, and Microscopical) required for Examinations in Public Health and to educate for the position of Public Analyst.

The Chemical Laboratory at Wiesbaden.—The Wiesbaden Chemical Laboratory is, since the death of its founder, Geh. Hofrath Prof. Dr. R. Fresenius, now directed by his sons, Prof. Dr. H. Fresenius and Dr. W. Fresenius, and his son-in-law, Dr. E. Hintz, quite in the same way as hitherto. In the Summer Term, 1897, there were fifty-four students on the books. Of these, forty-one were from Germany, three from England, three from Switzerland, two from Russia, two from Sweden, one from Spain, one from Finland, and one from Brazil. Besides the Directors there are engaged, as teachers in the establishment, Dr. med. G. Frank, Dr. W. Lenz, Dr. L. Grünhut, and architect J. Brahm. The assistants in the instruction laboratory were three in number, in the Versuchsstationen (private laboratories) twenty-four. The next Winter Term begins on October 15th. During the last term, besides the scientific researches, a great number of analyses were undertaken in the different departments of the Laboratory (Versuchsstationen) on behalf of manufacture, trade, mining, agriculture, and hygiene.

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All persons desiring to be admitted as workers, must send evidence of scientific training, qualification, and previous experience in original research, along with a statement of the nature of the investigation they propose to undertake. Further information, together with forms of application, can be had from the ASSISTANT SECRETARY, Royal Institution.

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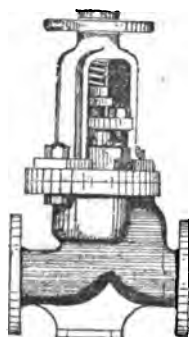
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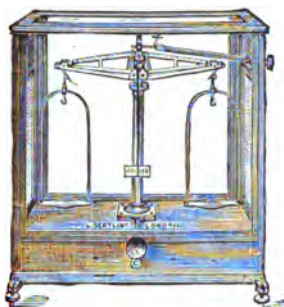
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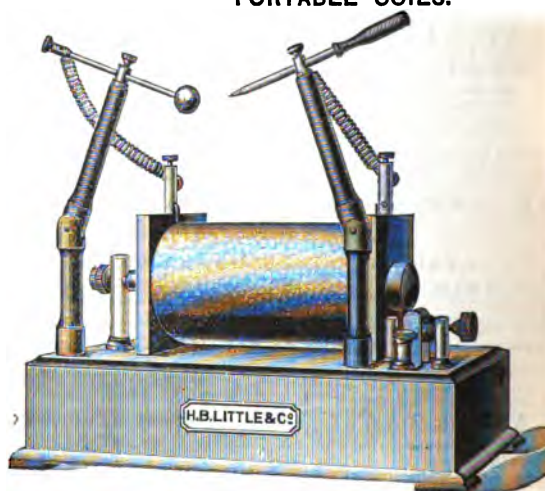
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THE CHEMICAL NEWS.

SEP 17 1897

VANADIUM IN RUTILE.

By W. B. GILES, F.I.C.

In the CHEMICAL NEWS (vol. lxxvi., p. 102) there appears a paper by B. Hasselberg entitled "Notes on the Chemical Composition of the Mineral Rutile," in which the following passage occurs:—"While the presence of vanadium in the rutile thus forms a *hitherto entirely unknown feature of this mineral*." I wish to point out that this is not correct, for rutile has been known to contain vanadium for over thirty years. Ste.-Claire Deville showed that this mineral contained not only vanadium, but also molybdenum. He says indeed in his paper, "Ou voit que le rutile est une matière dont on peut extraire le vanadium avec le plus grand avantage." ||

In 100 grms. of the rutile from Saint Yrieix he found—

0.323 grm. of vanadic acid, and
0.486 grm. of molybdic acid.

He also shows that vanadium is found in bauxite, in most clays, in cerite from Bastnäs, and in cryolite, as well as in the emerald and a variety of other substances. I may further point out that in these interesting researches he established the presence of tellurium and titanium, as well as vanadium in cerite, of tantalum acid in wolfram, and of niobic acid in cryolite. The methods he employed for the detection and estimation of the vanadium are very good, and probably no better way of effecting the separation of this widely-diffused element is known even at the present time. These researches are to be found in the *Annales de Chimie et de Physique*, 3me Series, t. lxi., and also in the *Comptes Rendus*, t. xlix., pp. 210 to 301.

ON A QUANTITATIVE SEPARATION OF ARSENIC FROM ANTIMONY.

By OSCAR PILOTY and ALFRED STOCK.

THE quantitative separation of arsenic, antimony, and tin is one of the most difficult problems of mineral analysis if the separation of each element is required.

We shall now describe a procedure which we have elaborated for the separation of arsenic from antimony, and which seems to us calculated to solve this question with accuracy and expedition. We have carried out this method quantitatively as regards arsenic and antimony. The separation from tin has been tried only qualitatively.

The observation upon which our method depends is the volatility of arsenic with hydrogen sulphide in a strongly hydrochloric acid solution. This fact may perhaps throw a light on the causes of some of the many discrepancies observed in the precipitation of arsenic as sulphide. If we heat arsenic tersulphide with very strong hydrochloric acid until there is a brisk discharge of hydrochloric acid, the chief part of the arsenic escapes with moderate ease, and the yellow arsenic sulphide disappears almost entirely. Indeed our experiments showed that from a solution of arsenic tetroxide or pentoxide no sulphide is precipitated by sulphuretted hydrogen if it is heated to ebullition with a simultaneous introduction of gaseous hydrochloric acid. Under these conditions the arsenic distils away entirely in a short time from the solution, probably as trichloride.

From the foregoing it will be readily seen that the precipitations of arsenic can be accurate only if the solutions

containing the metal are but slightly acid, or are not heated in presence of much hydrochloric acid.

We must emphasize this condition the more as we cannot find it mentioned either in journalistic literature or in standard works on chemical analysis. On the contrary, it has often been definitely shown that arsenic pentasulphide precipitates in heat from a hydrochloric solution, and contains tersulphide in abundance, in full contradiction with the statements of Bunsen concerning the precipitation of pentasulphide. Our observation seems to us fully to explain this contradiction.

The partial reduction of arsenic acid on heating in concentrated hydrochloric acid has been already noticed. Latterly Neher has reported on this observation. He informs us that under certain circumstances, on precipitating such a boiling solution with hydrogen sulphide, there is formed above the liquid a cloud of arsenic trisulphide. But he also failed to notice the behaviour of concentrated hydrochloric solution on the action of sulphuretted hydrogen, and the decomposability of the tersulphide already precipitated.

We should here remark that the precipitation of the arsenic in the slightly hot hydrochloric solution, as first used by Bunsen, always gives excellent results. The attributes of arsenic pentasulphide are so striking that the weighing of arsenic in this form seems preferable to all other methods.

Since other metals on the permanent presence of an excess of hydrochloric acid are not affected by hydrogen sulphide, it was our task to apply the described behaviour to arsenic for its quantitative separation from all other metals. We undertook, in the first place, its separation from antimony, and we obtained very accurate results.

We are now engaged with experiments to dispense with a double precipitation of arsenic.

We have hitherto practically effected only the separation of arsenic from antimony. We shall next report on the separation of arsenic from tin and the other elements of arsenic sulphide group.—*Berichte*, No. 12, p. 164.

ON THE PURIFICATION AND ATOMIC WEIGHT OF CERIUM.*

By MM. WYROUBOFF and VERNEUIL.

OF all the metals which we are in the habit of calling rare, cerium is without doubt the least known. A large number of researches have been devoted to it, and one would think in reading them that the subject must be well nigh exhausted. However, in taking the facts which seem the most incontestable one by one, we soon see that we are in a domain of uncertainty and contradictions. We only know the atomic weight of cerium approximately, we are uncertain as to its valence, and we are not even quite sure of its identity. Is it really a simple element, as we have hitherto attempted to believe, or does it consist of a group like the didymium of Mosander? like the erbium of Bahr and Bunsen? This last opinion has been upheld quite recently by the late M. Schützenberger in a series of important memoirs (*Comptes Rendus*, cxx., pp. 663, 962; cxxiv., p. 481). There should be, according to this eminent chemist, several elements presenting all the chemical and physical characteristics of cerium, and having atomic weights varying from 85 to 104 (Ce being taken as bi-valent). If such were the case, all the chemistry of cerium which has been done up to the present would have no longer any *raison d'être*, and every effort should be directed towards the separation of the different simple bodies of which it is formed.

We now extract, from a comprehensive research which will appear shortly, that which appears to concern the

capital question of the identity of cerium, for the purpose of showing that it is a definite, distinct body, possessing always—no matter what its origin may be—the same atomic weight, and incapable of being split up into more simple elements by any known process.

Two misconceptions dominate the whole chemistry of cerium; the one has regard to its separation from its neighbouring metals, the other to the determination of its atomic weight. It is these two misconceptions which appeared to us important to first of all clear up.

Purification of Cerium.—There are three impurities associated with cerium which are very difficult to get rid of. These are, in order of difficulty of elimination, iron, didymium-lanthanum-yttria, and thoria.

Iron.—It is generally believed that a precipitation of oxalic acid, or oxalate of ammonia, suffices to completely eliminate the iron. This is entirely erroneous. Two or even three precipitations, in warm and acid solution, hardly suffice to remove the last traces of iron. Its presence, even in minimal quantities, is manifested by the colour of the calcined ceroso-ceric oxide, which takes a more or less pronounced rose tint, sometimes even reddish.

Didymium and Lanthanum.—We have admitted, as a fact without question, that Debray's process—which consists, as is well known, in the twice-repeated fusion with nitre at about 320° —separates integrally the cerium from the other metals of its group. Nothing, however, is less certain. This process, which is purely empiric, appears to be based on the more and more difficult decomposition of the nitrates of protoxide of cerium, didymium, and lanthanum; in reality it is accompanied by a phenomenon infinitely more complex. The cerous nitrate passes firstly to the state of ceric nitrate, which easily gives an extremely stable basic salt, and this nitrate combines at a higher temperature with the nitrates of didymium and lanthanum to form a complex oxidised salt, to which we shall return later. We can follow this reaction by evaporating the red solution obtained by dissolving the calcined oxides in nitric acid to dryness, and heating to increasing temperatures. Towards 120° , when all the nitric acid is driven off, the mixture has a bright yellow colour. If at this moment we treat it with water, we obtain a pale yellow insoluble body, consisting of absolutely pure cerium in the state of nitrate $(\text{Ce}_2\text{O}_4)_4\cdot\text{N}_2\text{O}_5$, and a violet liquid containing cerium in the state of protoxide, with all the lanthanum and didymium. On reheating the mass, it gives off nitrous fumes and takes a chamois colour, which becomes accentuated as the heating proceeds; taken up with water, the mass gives an opalescent liquid which it is impossible to filter. This change of colour and of properties indicates that a new nitrate, of an oxide altogether different to Ce_2O_4 , is formed. In fact, in the presence of protoxides of more energetic basicity, the ceroso-ceric oxide tends to form an oxide, $\text{Ce}_3\text{O}_4\cdot 3\text{MO}$, which becomes remarkably stable at high temperatures when $\text{M} = \text{Di}$ or La . This oxide, which we propose to study in detail in a future note, gives very beautiful salts, which do not by any means resemble the yellow salts of the oxide Ce_2O_4 . It follows from this that Debray's process does exactly the opposite to what he proposes; instead of separating, it tends to combine Ce_3O_4 with $\text{DiO} + \text{LaO}$. No doubt by keeping the temperature up to 330° for a long time we can decompose this complex nitrate, but this decomposition is only achieved with great difficulty; it is for this reason that we can only arrive at a more or less complete separation, after a long series of fusions. It appeared to us to be much more rational to stop the reaction at the moment when the oxide Ce_3O_4 cannot combine with the $\text{DiO} + \text{LaO}$, for at this moment the cerium should be absolutely free, not only from the two other metals, but also from the metals of the yttria group which are always found in company with didymium.

Experience fully confirms this forecast, and we have been able to prepare in one day several hundred grms. of

perfectly pure cerium. This is how we set to work:—We calcine the oxalates gently, and treat them with nitric acid; here two different cases may present themselves.*

a. If the mixture contains more than 50 per cent of cerium, nitric acid will not dissolve it integrally, even with the aid of heat. One might be inclined to believe that the insoluble residue is formed of the pure oxide Ce_3O_4 , but such is not the case. It is a complex combination of the oxide Ce_3O_4 with $\text{DiO} + \text{LaO}$. In this case it is necessary to dissolve the oxalates in nitric acid, add an excess of peroxide of hydrogen and ammonia, and boil. The voluminous reddish brown precipitate of peroxide of cerium, and of the peroxides of Di and La which are formed, quickly lose oxygen, and become first orange-colour and then yellow. Having arrived at this state they then consist of ceroso-ceric hydroxide, $\text{Ce}_3\text{O}_4\cdot 3\text{H}_2\text{O}$, mixed with the protoxides of DiO and LaO . It only remains now to wash the precipitate to get rid of the nitrate of ammonia which interferes with the subsequent reaction, to dissolve it in hot nitric acid, and to continue the treatment according to b.

b. If the calcined oxides dissolve in nitric acid, we evaporate the solution to a syrupy consistency. It has a deep red colour, and contains cerium in the state of a salt of the oxide $\text{Ce}_3\text{O}_4\cdot 3\text{CeO} = \text{Ce}_6\text{O}_7$. To this semi-fluid mass we add a solution of 5 per cent nitrate of ammonia (thirty to forty times the weight of the oxides), and boil. If no precipitate is formed it is because the solution is too acid; we therefore add a weak solution of ammonia, drop by drop. Each drop will cause the formation of a flocculent violet precipitate, which re-dissolves on agitation up to the moment when a persistent pale yellow precipitate appears. When the supernatant liquor has no longer the least trace of yellow colour, but takes the characteristic violet colour of didymium salts, the reaction is at an end. The precipitate can be filtered and washed with the greatest ease, and when the wash water no longer gives a precipitate with oxalate of ammonium, the precipitate is absolutely free from lanthanum, didymium, and the earths of the yttria group. But the cerium thus obtained only represents a portion, about 75 per cent, of the total contained in the solution. It is not difficult to understand the reason. The action of nitrate of ammonium dissociates the oxide $\text{Ce}_3\text{O}_4\cdot 3\text{CeO}$, an oxide in which CeO is, by-the-by, partially replaced by DiO and LaO ; Ce_3O_4 is precipitated as nitrate $(\text{Ce}_3\text{O}_4)_4\cdot\text{N}_2\text{O}_5$, and CeO remains in solution with the other earths in the state of a neutral salt.

This process—the only one which enables us to obtain cerium completely free from lanthanum and didymium at one operation—can even be made to serve, as we shall show, for a sufficiently accurate method of qualitative separation. The cerium thus obtained, however, still contains an impurity from which it is not easy to separate it. At the same time as the cerium is precipitated, all the thorium in solution comes down also.

Thorium.—This metal, which is only just becoming known, and very little at present, nearly always accompanies cerium in its ores, even in cerite. All the ordinary methods in use will give us thorium free from cerium, but none of them give cerium free from thorium. Such is the case with the two best known of them, the hyposulphite of soda (Chydenius) and the suboxide of copper method proposed by Lecoq de Boisbaudran. We know that the former precipitates barely 85 per cent of thorina, and experiments made on synthetic mixtures have shown us that the latter removes only about 35 per cent. Furthermore, in both cases, a notable quantity of cerium is carried down, but this can be separated by repeating the operation two or three times. It is through not being sufficiently careful to guard against the presence of thorium, and through having accumulated it in successive fractional crystallisations, that we have sometimes found such

* When the oxides contain more than 10 per cent of thorium, it is as well to get rid of the greater part of it, by means of carbonate of ammonia, as will be shown further on.

strange variations in the atomic weight of cerium. The solution of the mixed sulphates of thorium and cerium, containing an excess of the latter, behave on evaporation quite differently to what one would be inclined to expect from the indications found in classic works. It is cerium, the most soluble, which separates out first: this goes to show that an excessively soluble double salt is formed, which crystallises with difficulty; it dries at the ordinary temperature to the condition of a transparent varnish. The analysis of this dehydrated salt leads us approximately to the formula $4\text{SO}_4\text{Ce}, \text{SO}_4\text{Th}$. At the ordinary temperature 100 parts of water dissolve 66 parts of the anhydrous salt.

The best way of getting rid of the thorium, when it is present in any quantity, is to treat the oxalates, or better still the nitrates, with carbonate of ammonium to which has been added a little ammonia. The thorium dissolves with the greatest facility, and after two or three leachings there is only an insignificant quantity (1 per cent) of thorium left.

To remove the last traces we make use of the property we have just mentioned, by crystallising the sulphate, free from free sulphuric acid, at 50° to 60° . The thorina remains in the mother-liquor, and after two or three crystallisations nitride of potassium—the most sensitive known reagent for thorium—will no longer give any precipitate. Cerium thus prepared may be considered as pure, at least within the limits of our actual knowledge, and when transformed into sulphate will serve for the determination of the atomic weight.

(To be continued).

A CRITICAL REVIEW OF THE METHODS OF DETERMINING MINERALS.*

By Dr. JOSEPH W. RICHARDS,
Of the Department of Metallurgy and Mineralogy of the Lehigh University.

(Concluded from p. 116).

I WILL end this review by describing the methods of determinative mineralogy taught at the Lehigh University, and which my ten years' experience has proven the most certain way of teaching a student to identify minerals.

First of all, the basis is the chemical identification, but with the aid of physical tests brought in at the most profitable stage of the inquiry.

The observer is to first examine the specimen carefully, noting everything that is to be seen or observed by the most simple tests, such as approximate hardness and streak, but not to spend any length of time, more than a minute or two, at such observation. The object of this casual inspection is to determine whether the mineral is similar to any that the observer has seen before; if it is, and therefore suggests one or even several species, the observer is immediately to make a confirmatory test, preferably chemical, and as general as possible, to see if it can possibly be what is suspected. If the test is affirmative, then other confirmatory tests must be applied, with the particular purpose of excluding all related minerals of closely allied composition. If the test is negative, and no other mineral or minerals suggest themselves, then the general method of procedure, based solely on chemical composition, is to be followed.

As an illustration, suppose a mineral appears on casual inspection to be wavelite. The first test is to determine whether it is a phosphate or not. If it is, then alumina, water, and finally fluorine, can be tested for to complete the identification. If not a phosphate, it may have been observed that the mineral glowed strongly and gave a calcium flame, which would suggest *aragonite*, and a test

for carbonic acid would be in order. Or it might have been observed that it fused easily, thus suggesting a radiated zeolite, and tests for silica and water would be in order. If none of these were observed, then the mineral would have to be attacked along broader lines; but if any of these tests have resulted affirmatively, then the observer has profited by his previous knowledge to obtain a short cut to the identification of the specimen. It is evident that the wider the observer's previous acquaintance with numerous varieties of numerous species, the more he will profit by his experience and be able to put it to use in identifying the specimen.

If the observer has no definite idea, from this casual inspection, of what the mineral may be, or if the ideas given by casual inspection were negated by the confirmatory tests, then some regular outline of procedure must be followed, and the following outline is offered as having been used very successfully for several years by your lecturer. Leaving aside the first three items, as being intended for identifying a few simple mineral species, clearing the ground, so to speak, the real classification begins at IV. All the species included in I., II., and III. are included in the later classes.

The outline is as follows:—

Lecturer's Methods.

- I.—If metallic and malleable, look for native metals.
 - II.—If very light and black, examine for hydrocarbons.
 - III.—If it has a taste, examine for chlorides, nitrates, sulphates, &c.
 - IV.—If of metallic, adamantine, or resinous lustre. test in the *open tube* for *S, As, Sb, Se, Te*. If present, roast thoroughly, and test by beads, flame, and reduction.
 - V.—If of any other lustre, or if no test is obtained in IV., *make bead test for silica*. If present, make flame test, &c.
 - VI.—If silica is absent, test for phosphates and borates. If test is obtained, make bead test, reduction, &c.
 - VII.—If P and B are absent, test for carbonates. If test is obtained, make flame test, beads, reduction, &c.
 - VIII.—If CO_2 is absent, test for sulphuric acid. If test is obtained, make flame test, beads, reduction, &c.
 - IX.—If SO_3 is absent, test with KHSO_4 for volatile acids. If test is obtained, make flame test, beads, &c.
 - X.—If volatile acids are absent, make test IV. (open tube), if it has not previously been made.
 - XI.—If open tube has already shown nothing, make the bead tests very carefully, to find the weaker acids, such as Cr, V, U, Ti, Mo. If present, make any other tests.
 - XII.—If weaker acids are absent, substance is probably an oxide. Make any blowpipe tests not already made.
- It will be observed that the outline follows generally the broad lines of the classification of mineral species used by Dana, the primary idea being to class the mineral first by finding its acid constituent. This has the disadvantage of bringing most of the oxides and native metals last in the classification, and makes it desirable that the observer be well informed with those classes by previous acquaintance, as thus much time is frequently saved. If there is any doubt about the lustre, the testing begins at IV.; if the lustre is certainly not of the kinds therein named, it is best to begin at once at V.
- In testing for the acid ingredient, those are tested for first which are most likely to be present (other things being equal), and also those tests are made first which are the quickest to make (other things being equal). Thus, silica is tested for before carbonic acid, because the silicates are more numerous; while phosphates are tested for before carbonates, because the test takes less time.
- After an acid is found, the direction of the testing is towards the basic ingredient. It should be observed, however, that if the tests at any time in any way such information about the mineral that the observer suspects what it is, then let him at once leave the general outline,

* The Journal of the Franklin Institute, cxliv., p. 139.

and by direct tests confirm or deny his suspicion. If, for instance, when testing for phosphorus the barium flame is seen, and the observer then looks at the mineral again and suspects *barite*, then he should test at once for sulphuric acid to affirm or negative his suspicion, without going through the form of testing for carbonic acid. If sulphuric acid is absent, then let him take up the scheme again where he left it, and test in regular order for carbonic acid.

By following these directions, the composition of a mineral, or at least one acid and one base present, can quickly be found. The question then arises, how many minerals can contain the ingredients so far found; and in order to facilitate the search for minerals containing certain elements, I have prepared tables of minerals arranged according to the elements they contain. These tables contain under the heading of each element a classification of all the minerals containing that element, arranged primarily according to the acid ingredients, in the order in which they would be found when following the outline of procedure I have indicated. Under each acid the minerals are further classified according to the other constituents which may be present, arranged in the order in which they are most quickly and surely identified by blowpipe tests, followed by these minerals containing only the acid and the base, or possibly containing some other ingredients not recognisable by blowpipe tests. Wherever, as often happens, a list is obtained of several minerals containing exactly the same ingredients, a small table of physical properties placed to the right gives the means of distinguishing them apart. A sample page is as follows:—

LECTURER'S TABLES (sample page).

Copper.

Physical Properties,
Composition, &c.

Sulphates:—

With Pb:

- † Cl: Caracolite.
- † Fe: Beudantite.
- : Caledonite, linarite.

With Zn:

- † Al: Aluminite (impure).
- : Serpierite.

With Fe:

- † Al: Cyanotrichite.
- : Phillipite, pisanite.

With U:

- † Ca: Uranochalcite.
- : Voglianite, johannite (H_2O —5 per cent).
- : Zippelite (H_2O —16 per cent).

With Na:

With K:

With Ca:

With Cl:

With H_2O :• :
• :

It will be observed that, granting that the experimenter has found sulphuric acid and copper, if he does not at once identify the specimen by that information, his next step should be to reduce with soda for lead and zinc; if lead is found, then tests for chlorine or iron are in order; and if neither of these be present, the mineral must be *caledonite* or *linarite*, which must be told apart by reference to their physical properties. If the reduction with soda has shown nothing, the bead test for iron and uranium is in order. If these are absent, the flame test for sodium, potassium, and calcium is to be made; and if these are absent, the special test for chlorine, and then water. If none of these are present, the mineral will be found in the list following the double asterisks, and distinguished in that list by its physical properties. (The names of these latter minerals and the physical properties are omitted in this sample page for lack of space).

It will be objected to this method that with minerals

which may contain small amounts of many ingredients their classification will be very difficult. This is admitted; but by classifying these minerals under each heading that they can possibly come, as well as under that heading where they must invariably come, the careful observer cannot fail to identify them, no matter what known variety of composition they may show.

The essential idea of this method of working is to use the physical properties as suggestive or confirmatory—*suggestive* on casual inspection of what the mineral may be, such suggestion to be confirmed by chemical tests; *confirmatory* after a determination by chemical tests. Conversely, the chemical properties or composition are used as the true *determinative* factors, to which the physical tests are subsidiary or confirmatory. Several years' practical use in the mineralogy laboratory has demonstrated, as far as my experience goes, the superiority of this method of determination, both in reliability and—taking the average—in quickness.

(At the close of the lecture there was shown a portable specific gravity hydrometer, made by Wood and Comer, of Philadelphia, with especial reference to the use of the mineralogist, and which gives rapidly and accurately the specific gravity of pieces weighing not over 1 grm. Its use in the laboratory work at Lehigh University has demonstrated this claim, and shown that it meets the present need for a cheap, portable, and accurate instrument for determining specific gravities).

THE ATOMIC MASS OF TUNGSTEN.*

By WILLETT LEFLEY HARDIN.

A REVIEW of the literature on the determinations of the atomic mass of tungsten will show that a careful examination of the methods employed is of fundamental importance. Fifteen experimenters have made determinations of this constant, but with widely varying results. The method of investigation, with few exceptions, has been to reduce tungsten trioxide in a current of hydrogen at a white heat, and then to re-oxidise the metal thus obtained. Deviations occur not only in the results of the different experimenters, but also, with few exceptions, in the different observations of the same experimenter. Clarke ("A Re-calculation of the Atomic Weights, 1897") closes his summary of all the work on the atomic mass of tungsten with the following words:—"Further investigation is required in order to fully establish the true atomic weight of tungsten."

Deviations in the results of any atomic mass determinations are due either to an inaccurate method or to experimental errors. It is not always an easy matter, however, to determine the source of error in a given series of variable results; but with several series of results from different experimenters, the question can be decided with a reasonable degree of accuracy. If, for instance, we have several series of results by the same method from different experimenters, and if the different series, with the exception of one, agree with each other, the probability is that the deviations in the one series are due to experimental errors. If, on the other hand, deviations occur in each series, and are more or less similar, the probability is that the method is inaccurate.

Almost every series of results on the atomic mass of tungsten, obtained by the reduction of the trioxide in a current of hydrogen, and by the re-oxidation of the resulting metal, shows a variation between the maximum and minimum results of from one to two units, and in exceptional cases the deviation is much greater. In view of these facts, it seems desirable to make a careful study of the method which has usually been employed in this work, rather than add to the already large number of re-

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xix., No. 8.

sults; for, if the method is unreliable, no experimental skill can make the results trustworthy.

The following summary will show the lack of concordance in the results of the earlier determinations:—

Berzelius (*Pogg. Ann.*, viii., 1, 1826) was the first to determine the atomic mass of tungsten. By reduction of the trioxide he obtained the value 189.6 as a mean of two experiments for the atomic mass of tungsten. The difference between the two results was 3.0. These results and those that follow are calculated on the basis of O = 16.

Schneider (*Fourn. Prakt. Chem.*, 1., 152, 1850), working with material which had been carefully purified, obtained two series of results for the atomic mass of tungsten, one by the reduction of tungstic acid and the other by re-oxidation of the metal.

Reduction series.	Oxidation series.
184.18	184.21
183.37	184.16
184.01	183.36
184.28	
184.45	Maximum diff. .. 0.85

Maximum diff. .. 1.08

The quantity of material used in these experiments varied from 2 to 6 grms.

Marchand (*Ann. Chem. Pharm.*, lxxvii., 261, 1851) reduced the trioxide of tungsten and re-oxidised the resulting metal; the following results were obtained for the atomic mass of tungsten:—

183.91	} Reductions.
183.96	
184.16	} Oxidations.
184.51	

Maximum difference .. 0.60

Borch (*Fourn. Prakt. Chem.*, liv., 254, 1851) made seven reductions of tungstic acid in a current of hydrogen, and two oxidations of the metal. The results were as follows:—

Reductions.	Oxidations.
184.10	184.53
182.90	184.32
183.77	
184.10	Difference .. 0.21
183.03	
183.77	
183.91	

Maximum diff. .. 1.20

The quantities of material varied from 2 to 8 grms.

By weighing the water obtained in the reduction of tungstic acid in hydrogen, Riche (*Fourn. Prakt. Chem.*, lxxix., 10, 1857) obtained the value 174 for the atomic mass of tungsten as a mean of two experiments. The difference between the two was 1.78.

Dumas (*Ann. Chem. Pharm.*, cxiii., 23, 1860) reduced the trioxide of tungsten in hydrogen and obtained the following results for the atomic mass of tungsten:—

184.00
183.42
184.16
183.76
183.62
184.80
184.16
184.08

Maximum difference = 1.38

The quantities of material varied from 1 to 4½ grms. Bernoulli (*Pogg. Ann.*, cxl., 573, 1860) reduced tungstic acid at a very high temperature in a current of hydrogen. The results were as follows:—

Reduction series.	Oxidation series.
186.78	186.81
185.86	187.94
186.75	186.77
186.81	186.76
186.70	
177.73	Maximum diff. = 1.18

Maximum diff. = 9.08

The maximum difference in the two series is 10.21. He found that the greenish coloured oxide gave the same results as did the yellow oxide.

Persoz (*Ann. Chim. Phys.*, [4], i., 93, 1864) made two reductions of the trioxide of tungsten and obtained concordant results.

183.93
183.94

Difference = 0.01

Scheibler (*Fourn. Prakt. Chem.*, lxxxiii., 324, 1861), from determinations of the water in barium metatungstate, obtained the value 184.00 for the atomic mass of tungsten. Maximum difference = 1.03.

Zettnow (*Pogg. Ann.*, cxxx., 30, 1867) obtained the value 184.08 for the atomic mass of tungsten as a mean from four analyses of the tungstate of iron. From silver tungstate he obtained the value 183.80.

Roscoe (*Ann. Chem. Pharm.*, clxii., 368, 1872) made three reductions and two oxidations of the same sample of material, beginning with 7.8840 grms. of tungstic acid. The results were as follows:—

Reductions.	Oxidations.
182.72	182.49
183.71	183.87
183.97	

Difference = 1.38

Maximum diff. = 1.25

From two analyses of tungsten hexachloride, Roscoe obtained the value 184.25 for the atomic mass of tungsten.

Waddell (*Am. Chem. Journ.*, viii., 280, 1886), from carefully purified tungstic acid, obtained by reduction in hydrogen the following values for the atomic mass of tungsten:—

184.55
184.37
184.59
184.00
183.67

Maximum difference = 0.92

The quantities of material varied from 1 to 4½ grms.

The material used in the work of Pennington and Smith (*Zeit. Anorg. Chem.*, viii., 198) differed from that of all the preceding experimenters, in that the last traces of molybdenum were removed by gently heating the tungstic acid in a current of hydrochloric acid gas. The method of operation was also somewhat different from those of the earlier experimenters. The metallic tungsten used in the oxidations was obtained by the reduction of tungstic acid in a platinum crucible at a white heat, in a current of hydrogen, which was conducted through the lid of the crucible. The mean of nine results from the oxidation of the metal is 184.921 for the atomic mass of tungsten. The maximum difference in the series is 0.043. The quantities of material used varied from 0.43 to 1.08 gm.

Smith and Desi (*Zeit. Anorg. Chem.*, viii., 205) weighed the water obtained in the reduction of tungstic acid, and from that calculated the atomic mass of tungsten. The mean of six determinations is 184.704. Maximum difference 0.071.

Schneider (*Fourn. Prakt. Chem.*, liii., 288, 1896) made a second series of reductions and oxidations. The material used in these experiments was freed from molybdenum by gently heating the tungstic acid in a cur-

rent of hydrochloric acid gas. The values obtained for the atomic mass of tungsten were as follows:—

Reduction series.	Oxidation series.
184.14	184.00
183.98	183.92
183.96	184.04

Maximum diff. = 0.18 Maximum diff. = 0.12

The quantities of material varied from 2 to 6 grms.

Shinn (Thesis, University of Pennsylvania, 1896) obtained by oxidation of metallic tungsten the following values for the atomic mass of tungsten:—

184.72
184.96
184.75
185.22

Maximum difference = 0.48

The quantities of material used varied from 0.10 to 0.22 gm. of metal.

A glance at the foregoing results will show a remarkable variation. The extremely high value obtained by Berzelius is supposed to be due to the presence of alkaline impurities in the material used.

The observations of Schneider, Marchand, Borch, Dumas, and Waddell are very similar. The method of operation was the same in each case, and the material used was purified with considerable care. It is difficult to account for the variations which occur throughout these results. Molybdic acid is probably the only impurity that could have contaminated the material used in the experiments. Such an impurity would probably have lowered the separate results by the same amount, and hence would not have produced the variations. The deviations between the maximum and minimum results of the different experimenters are as follows:—Schneider 1.08, Marchand 0.60, Borch 1.63, Dumas 1.38, and Waddell 0.92.

The results of Riche and Bernoulli differ widely from those obtained by other experimenters. The extremely low value obtained by the former is probably due to the method. The high results obtained by Bernoulli are more difficult to explain. The material used was carefully purified. The tungstic acid used in some of the experiments was of a greenish tinge. Some have assumed that this material was incompletely oxidised, and in this way account for the high results. Bernoulli found, however, that the greenish coloured oxide and the yellow oxide gave the same results when reduced. Furthermore, neither the colour of the original oxide nor the state of oxidation could affect the results obtained in the re-oxidations of the metal. The results obtained by the latter method are higher than those obtained in the reductions. In view of these facts, the explanation which has been offered to account for these high results is entirely unsatisfactory.

Scheibler's results on barium metatungstate show a variation of more than one unit, and it must be added that the results obtained by the determination of the barium and tungsten in this salt were still more variable and were not used by Scheibler in calculating the atomic mass of tungsten. The two short series of results on ferrous and silver tungstates by Zettnow are reasonably concordant.

Roscoe's experiments on the same sample of material are rather interesting. The material was reduced and re-oxidised several times without being removed from the porcelain boat. The maximum difference in a series of five results is $\frac{1}{4}$ units. If the method employed by Roscoe is accurate, it is difficult to account for this variation.

The most concordant series of results on the atomic mass of tungsten is that of Pennington and Smith. The value obtained is higher than that obtained by most ex-

perimenters. Schneider (*Journ. Prakt. Chem.*, liii., 283, 1896) has attempted to account for the high values obtained in these experiments; but, inasmuch as these results agree very closely with those obtained by Smith and Desi and Shinn, it is useless to offer an explanation for this high value until the true atomic mass of tungsten is known with greater certainty, at least until a series of concordant results has been obtained which differs from these.

Schneider's last determinations consist of two series of results, each series containing three observations. From these two short series of reasonably concordant results, Schneider concludes that the atomic mass of tungsten may be safely considered equal to 184.00. The evidence, however, is far from satisfactory. In view of the wide variations in the earlier determinations, the number of results in these experiments is entirely too small to establish anything with certainty with regard to the true atomic mass of tungsten. This fact is shown in the work of Waddell, who made five determinations. The maximum variation in the first three observations was only 0.22, while in the series of five the variation was 0.92. The same is noticed in the work of other experimenters. And in the present investigation, consisting of more than sixty determinations, a series of five concordant results were sometimes obtained, after which considerable variation was obtained. Attention will be called to this fact again in the discussion of the following observations.

(To be continued).

EXPERT TESTIMONY.*

By WILLIAM P. MASON.

It will be remembered that a would-be facetious barrister once remarked that prevaricators might be properly arranged in an ascending series, to wit, ordinary fibbers, liars, and experts,—an arrangement which I fear meets with the approval of many members of the bench and bar to-day. The cause for such harsh classification is not so very far to seek. It is based upon ignorance on the part of the bar, and at times upon what is worse than ignorance on the side of the "expert." With the culpable acts of the pseudo scientist we cannot waste our time. That he merits prompt condemnation is axiomatic; but a word is wanted touching upon what may be termed the ignorance of the court.

"When I take my place upon the witness stand," said a prominent toxicologist once to me, "I can never predict in what shape I shall be upon leaving it," a feeling with which most of us can, I fancy, sympathise pretty keenly.

Is it that we fear exposure of the weak points in our professional armour? Do we dread to say in public "I do not know?" Hardly that, I take it. We are now possessed of so very little of that which one day may be known, that no true scientist hesitates for an instant to plead legitimate ignorance. What really troubles us upon cross-examination is that the court does not speak our language, a language often quite difficult of direct translation; that it is but rarely schooled in the principles of our science; and that, in consequence, it frequently insists upon categorical answers to the most impossible kind of questions.

The hypothetical questions showered upon the expert witness are sometimes veritable curiosities, so peculiar are they in their monstrosity. Who among us but has felt that the layman, who has simply to testify to observed facts, has an easy time of it indeed, when compared with him from whom there is expected an opinion under oath?

* An Address by Vice-President William P. Mason, Chairman of Section C, before the Section of Chemistry, American Association for the Advancement of Science, Detroit Meeting, August, 1897.

All scientific men are willing and anxious to have their work scrutinised carefully by their peers; but to be exposed to the one-sided criticism frequently encountered at the bar is quite another matter; for it must be remembered that after the adverse counsel has opened up what appears to be a glaring inconsistency in the testimony, the re-direct examination may utterly fail to repair the breach, because of a lack of familiarity with a technical subject on the part of the friendly attorney.

This leaves the witness in the unenviable position of disagreeing with the general drift of his own testimony, while it deprives him of suitable means of insisting upon its revision and correction.

According to the writer's view there is but one way to escape such dilemma, and that is by direct and immediate appeal to the judge, urging that the oath taken called for a statement of the whole truth, and not the misleading portion already elicited.

To illustrate how serious a matter the partial testimony of an expert witness may be, and to show also to what extent lawyers may go who look only to the winning of their causes, permit me to refer to an already reported poison case in which I was employed by the people. It may be roughly outlined as follows:—

Much arsenic and a very little zinc were found in the stomach.

The body had not been embalmed, but cloths wrung out in an embalming fluid containing zinc and arsenic had been spread upon the face and chest.

Medical testimony showed that no fluid could have run down the throat. Knowing the relative proportions of zinc and arsenic in the embalming fluid, the quantity of arsenic found in the stomach was twelve times larger than it should have been to have balanced the zinc also there present, assuming them to have both come from the introduction of the said embalming fluid by cadaveric imbibition. Other circumstantial evidence was greatly against the prisoner.

At the time of my appearing for the people, on the occasion of the first trial of the case, my direct testimony brought out very strongly the fact that a fatal quantity of arsenic had been found in the stomach, but no opportunity was given me to testify to the presence of the zinc found there as well, although the fact of its existence in the body was known to the prosecution through my preliminary report. Through ignorance of the nature of such report on the part of the defence, no change was made in the character of my testimony during the cross-examination, and I was permitted to leave the witness-stand with a portion of my story untold. No witnesses were called for the defence, and the case was given to the jury with the darkest of prospects for the prisoner.

For many reasons, unnecessary to recount here, I was distinctly of the opinion that murder had been committed, but I felt nevertheless that common justice demanded that the prisoner should have been entitled to whatever doubt could have been thrown upon the minds of the jury, no matter how far-fetched the foundations for such doubt might have been.

The first trial having resulted in a disagreement of the jury, I was pleased to learn, before the second hearing of the case began, that the defence was prepared to go into the question of the embalming fluid; for the responsibility of permitting only a part of what I knew to be drawn from me, to the entire exclusion of the remaining portion, was greater than I wished to assume. The nature of my report to the coroner having been established, and certain opinions relating thereto having been fully ventilated, the jury were possessed of "reasonable doubt" and acquitted the prisoner. What now were the duties of the expert upon the occasion of the first trial of this case, and how should he have construed the meaning of his oath?

One eminent legal light, to whom the question was referred, held that the expert was distinctly the property of the side employing him, and that his duty was simply to answer truthfully the questions put to him, without at-

tempting to enlighten the court upon facts known to him, but not brought out by the examination, no matter how vital such facts might be.

Another held that although the above course would be proper in a civil case, yet, in a matter involving life and death, the witness should insist upon the Court becoming acquainted with his whole story. Do not such differences in legal opinion make it very desirable that the expert, at least in capital cases, should be the *employé* of the bench rather than of the bar, in order that whatever scientific investigations are made may be entirely open to public knowledge and criticism?

Although the expert should earnestly strive to have what he has to say presented in the best form, he must remember that to secure clearness, particularly before a jury, technicalities should be reduced to a minimum. To a degree they are unavoidable, but let them be as few as possible. Illustrations should be homely and apt, capable of easy grasp by the jury's minds, and, if possible, taken from scenes familiar to the jury in their daily lives.

It is an unfortunate fact that the expert must be prepared to encounter in the Court-room not only unfamiliarity with his specialty, but also deep-rooted prejudices and popular notions hoary with age and not to be lightly removed from the mind by the words of a single witness. As President Jordan has well said, "There is no nonsense so unscientific that men called educated will not accept it as science," and, let me add, they will calmly attempt to shove the burden of proof upon the scientific man who is opposed to their views. Sanitary experts, in particular, run up against all sorts of popular superstitions, and are inveighed against as "professors" by those who consider themselves the "practical" workers of the time; and, let it be noted, the burden of proof is uniformly laid upon these "professors" shoulders, while the most astounding and occult statements made by the "practical" men may be received without verification.

One source of trouble, which perhaps is peculiar to the water expert, lies in the impossibility of utilising analytical results such as were made many years ago.

Those who are not chemists fail to grasp the fact that the examination of water may not be looked upon from the same point of view as the analysis of an iron ore. The statement that water analysis is but of recent birth, and that it is yet in its infancy, is hard for them to appreciate, holding, as they naturally do, that what was true twenty years ago must be true to-day, if science does not lie.

A pit into which many an expert witness falls is prepared for him by insidious questions leading him to venture an opinion upon matters outside of his specialty. It is a fatal error to attempt to know too much. Terse, clear answers, well within the narrow path leading to the point in question, are the only safe ones; and when the line of inquiry crosses into regions where the witness feels himself not truly an expert, his proper course is to refuse to testify outside of the boundaries of his legitimate province.

Unfortunately the expert is as often invited to take these collateral flights by the side employing him as by the opposition. Affidavits in submitted cases are commonly written by the lawyers and not by the expert, although they are, of course, based upon his reports. In the strength of his desire to win the case, the lawyer often prepares a much stronger affidavit than his witness is willing to swear to.

The writer has had no little difficulty just at this point, and has had plenty of occasion to observe the irritation displayed by counsel upon a refusal to endorse statements which have been "too much expanded."

Every expert-witness, especially in his early cases, is sure to have adverse authorities quoted against him; therefore it behoves him to be so familiar with the literature of his subject as to be capable of pointing out that such and such a writer is not up to date, or that such and such a passage, if quoted in full, would not bear the ad-

verse construction that its partial presentation carries. When the expert reaches a position of such prominence that he can state a thing to be so because he says it, irrespective of whatever may be written on the subject to the contrary, his course then is greatly simplified; but long before he attains that altitude he will have put himself upon record in many cases, and happy for him if the record so made be such as cannot be quoted to his disadvantage.

"If I had only not written my first book," is the reflection of many a distinguished author; while one of the great masters of music, referring to an opera, said "It is one of my early crimes."

Above all things, the expert "should provide things honest in the sight of all men."

It is well for him to be deeply interested in his case, to feel in a measure as if it were his own; but it is unwise in him to become so partisan as to let his feelings affect his good judgment, and it would be indeed criminal should he permit his interest in any way to contort the facts.

Before the case is brought to a final hearing it may be apparent that experiments before the Court are possible, and they may be demanded by the counsel in charge of the case. If such experiments be striking, easy of execution, and not too long, by all means make them.

Practical illustrations, particularly such as involve some fundamental principle, have great weight with the Court; but these illustrations must not be such as would turn the Court-room into a temporary laboratory and involve the loss of much time in vexatious waitings.

Such experiments as are determined upon should be thoroughly rehearsed beforehand, no matter how simple they may be; for, of all failures, the Court-room experiment which declines to "go off" is perhaps the most dismal.

This brings to mind a kindred topic upon which there should be a word of caution: laboratory experiments which work to perfection may utterly fail when expanded to commercial proportions, so that it is wise to bear in mind the danger of swearing too positively as to what will happen in large plants, when the opinion is based only upon what is observed to occur upon the smaller scale. Like conditions will, of course, produce like results; but it is marvellous how insidiously unlooked-for conditions will at times creep into one's calculations, and how hard it is even to recognise their presence.

When preparing his case for presentation, the expert often errs in not dwelling more largely upon certain points because he thinks them already old and well known. To him they may be old, but to the public they may be of the newest. Not only is the public unequally posted with the specialist, but what it once knew upon the subject may have been forgotten. It is well therefore to insert, in a special report, matters that would be properly omitted from a paper prepared for a professional audience.

Sanitary problems are of especial interest to the public, but the amount of ignorance, or rather false knowledge, displayed concerning them is surprising and often difficult to combat. The sanitarian is not unfrequently called upon suddenly to defend a position involving complex statistics; and because the data cannot be forthwith produced, the inference is drawn that his points are really without facts to support them, and that they are consequently not well taken.

Long before he gets into court, particularly if the time for preparation of the case be short, the expert may well "pray to be delivered from his friends." He may receive a peremptory order by telegraph to "determine the mineral qualities of this rock," when the telegram should have read "Assay this ore for silver," and later it may be a matter of surprise that a quantitative knowledge of the copper present was not obtained while passing along the line for the determination of the silver; for it is generally not known that the complete analysis of anything is quite rare and correspondingly tedious and expensive.

Toxicologists who hear me may call to mind some case

involving a search for the presence of an alkaloid, strychnia for example, during which search the district attorney, in his eagerness for information, may have asked to know what the indications were as to the presence of the poison, at a time when the extraneous organic matter was not nearly removed. He may have wished no final report, but only the simple probabilities, whereon to base a possible arrest. Such requests are very common, and are akin to a demand for a proof of the pudding during the early baking, when we all know that such proof comes at a much later stage of the proceedings.

Finally, "When doctors disagree who shall decide?"

This question is often very vigorously settled by the jury, as was instanced in a recent celebrated murder trial in New York city. In that case what the experts had to say on either side was simply thrown overboard as a whole, and the finding was based upon the testimony of the remaining witnesses.

What can be said upon this question of the disagreement of expert witnesses? First, it must be noted, they are far from being the only class of people who fail to agree, and that too on very important subjects. Do my hearers think it would be a very difficult task to find a small army of men who would testify very variously and very positively upon questions of politics or religion? Would it be hard to find "good men and true" who would give under oath greatly differing opinions concerning the propriety of instituting free trade or establishing an inheritance tax? Experts are subject to the same errors of judgment as befall the rest of professional humanity, and, when their opinions clash, they are entitled to the same respect that we grant to the members of the Bench when they hand down the decision of a divided court.

One fruitful opportunity for disagreement always arises when questions are brought into court touching upon matters newly discovered, and apart from the well-beaten path of common professional knowledge. Doubt is often left upon the minds of those seeking the light, even when the testimony is given by the specialist who originally developed the new point in question, for one cannot be expected to be thoroughly educated in that which he has himself but recently discovered.

Many of us have dreaded to see the "ptomaines," or putrefactive alkaloids, make their way into court with their mystifying influences upon judge and jury and their tendency to protect crime. Now that they are in, what is to be the end? Even with no "ptomaine theory" possible, the ptomaine form of argument is not unknown. The writer was once asked, in an arsenic case, whether he was willing to swear that at some future time an element would not be discovered giving the stated reactions now called arsenical? Such nonsense is of course instituted to impress the jury, and is suggested by similar questioning in the alkaloid cases.

A recent and somewhat amusing instance arose from an attempt to introduce the rather new conception of "degeneracy" into a murder trial. The defence sought to show that the prisoner was a "degenerate," and offered expert testimony as to the meaning of the term and as to the signs whereby such a condition was to be recognised; whereupon the prosecution called attention to the fact that the defendant's experts themselves exhibited every one of the signs in question.

Having said all that he was to say, and having stated it to the best advantage, should the expert depend upon the stenographer's so recording it as to allow of its being used in future without correction? Decidedly not.

The average stenographer is unfamiliar with technical terms, especially such as are chemical, and the witness who fails to supervise the minutes may find out later that he was sworn to a most remarkable array of "facts." The writer once discovered that he had recommended, as a very efficient method of purifying a city water, the filtering of the entire supply "through a layer of black mud." Not to take your time further, let us summarise what has thus been briefly said:—

The expert witness should be absolutely truthful, of course; that is assumed, but beyond that he should be clear and terse in his statements, homely and apt in his illustrations, incapable of being led beyond the field in which he is truly an expert, and as fearless of legitimate ignorance as he is fearful of illegitimate knowledge.

Mounting the witness-stand with these principles as his guide, he may be assured of stepping down again at the close of his testimony with credit to himself and to the profession he has chosen.

NOTICES OF BOOKS.

Organic Chemical Manipulation. By J. T. HEWITT, M.A., D.Sc., Ph.D., F.C.S. (London and Berlin), Professor of Chemistry in the East London Technical College. With 63 Illustrations. London and New York: Whittaker and Co. 1897. Pp. 260.

It is very satisfactory to find the evidently increased attention now paid to organic chemistry, both in its analytical and its synthetical phases. Dr. Hewitt, in the work before us, is contributing ably to meet the demand thus springing up.

The first chapter discusses the purification of organic substances. In the second we come to the ultimate analysis of organic compounds, the determination of equivalent and molecular weights; whilst the fourth chapter treats of the estimation of special groups of atoms in organic compounds.

The second part enters upon the preparation of organic substances, classified under the heads of "Compounds of the Fatty Series" and "The Aromatic Hydrocarbons and their Derivatives."

After a careful inspection, we feel fully warranted in recommending this little work to the earnest student.

Manueli Hoepfli. Metallic Alloys and Amalgams; Aluminium, Nickel; the Precious Metals and their Imitations; Bronze, Brass, Coins, Medals, Solders. By the Engineer S. GHERSI. Milan: Ulrico Hoepli.

We find here a very complete account of alloys in general, their composition and properties. Special attention has been paid to the influence—generally deleterious—of traces of foreign metals. The tin of Banca approaches very closely to chemical purity, that of Queensland taking the second place. The coppers of Australia and of Lake Superior differ little in their degrees of purity.

The following alloy exhibits a curious modification of colour; it is purple with ruby-red reflections. The compound of 22 per cent of gold with 78 per cent of aluminium—known as Margot's alloy—is perhaps the most beautiful, but it is deficient in malleability. Margot is of opinion that its colour is due to microscopic crystals of alumina disseminated in the mass; but this is open to doubt. The yellow alloy of platinum and aluminium can, by a modification of the proportions, be rendered violet or greenish.

The experiments of Krupp are in favour of the use of iron-nickel for artillery and metal plating. If steel is of an inferior quality it is not improved by the addition of nickel.

A rose-coloured alloy is obtained with 750 parts gold, 200 parts silver, and 50 parts of copper.

Tables for Gas Analyses, Gas Volumetric Analyses, Determination of Nitrogen, &c. ("Tabellen für Gas Analysen, Gas Volumetrische Analysen, Stick Stoffbestimmungen, &c.") By Prof. Dr. G. LUNGE. London, Edinburgh, and Oxford: Williams and Norgate. 1897.

A SET of useful analytical tables, conveniently arranged for laboratory use.

CORRESPONDENCE.

OCCURRENCE OF VANADIUM.

To the Editor of the Chemical News.

SIR,—*Apropos* of the description of the occurrence of vanadium in rutile by Hasselberg (see CHEMICAL NEWS, vol. lxxvi., p. 112), the following analysis of a sample of rock from South Africa may be of interest. It was sent here for analysis as a sample of iron ore:—

SiO ₂	..	..	..	0.99
Al ₂ O ₃	..	..	..	2.06
Fe ₂ O ₃	..	..	..	72.85
FeO	..	..	..	5.24
MnO	..	..	..	0.20
CaO	..	..	..	0.40
MgO	..	..	..	0.54
TiO ₂	..	..	..	15.00
V ₂ O ₅	..	..	..	0.59
NiO	..	..	..	0.30
Cr ₂ O ₃	..	..	..	0.26
Combined H ₂ O	..	..	..	1.37
Moisture	..	..	..	0.58
Phosphorus	..	..	..	nil
Sulphur	..	..	..	trace
Arsenic	..	..	..	nil

100.38

—I am, &c.,

F. W. DAW, A.R.C.Sc., F.C.S.

Elbow Vale, September 4, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 7, August 16, 1897.

Dr. Ferrand made, through the mediation of Armand Gautier, a claim of priority as regards vaccination against cholera.

Researches on the Simple Cathodic Rays.—H. Deslandres.

Action of Röntgen Tubes behind Screens Opaque to X Rays.—Abel Buguet.—The radiations concerned do not come essentially from the solid bodies employed in the experiments. They are attenuated when the sensitive surface is more or less masked by sheets of paper, aluminium, tin, silver, or lead. They did not traverse a sheet of lead of 0.5 m.m. The radiations may be used in the radiography of partially opaque bodies in cases where the direct action of the X rays is arrested by impassable obstacles. The phenomenon is not affected by a magnetic field, nor by a powerful current of air playing over the surface of the sensitive body.

Bulletin des Travaux de la Société de Pharmacie de Bordeaux. July, 1897.

Analysis of the Oil of American Black Walnuts (*Juglans nigra*, L.).—MM. Barthe and Boutineau.—The tree producing these nuts was imported into Europe in 1656. It is now common in the Gironde and Lower Pyrenees, where it grows to a considerable size. And although the nut itself is not so agreeable to the taste as the ordinary walnut, on account of its slight flavour of turpentine, it gives a useful and quickly-drying oil. One

kilogram. of the kernel gives, as a rule, 470 grms. of oil; its specific gravity is 0.929 at 15° C.; its acid index, expressed in m. grms. of caustic potash, is 4.12; its index of saponification is 195; its index of refraction is +26° at 15° C. in Jean's refractometer; and it dissolves in absolute alcohol to the extent of 6 grms. per litre.

Examination of the Official Solution of Perchloride of Iron.—E. Falières.—This solution should contain 26 per cent of anhydrous ferric chloride, or 8.96 per cent of iron; its density is then 1.26. It may, however, be adulterated and its density then brought up to the right point by the addition of salts of soda or potash. The author has sought for a rapid method of estimating its purity, and found that this could be best done by alkalimetric titration of the quantity of acid in combination with the iron, and the estimation of the chlorine by nitrate of silver.

Possible Manner of the Formation of the Fossil Phosphates of Lime in the Province of Oran.—M. Dion.—The author concludes that these fossil phosphates have an aqueous origin; that they are formed, from above, downwards, by the action of infiltrating rain-water; and that animal remains were the only source of phosphorus in these deposits, as no phosphates belonging to the crystalline minerals are to be found in the neighbourhood.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xvii.-xviii., No. 13.

M. Gautier presented, on behalf of M. Hélier and himself, the researches they have carried out, on the combination of chlorine and hydrogen under the influence of light and heat.

M. Villiers pointed out a method of oxidation of minerals, and of organic compounds of the fatty series; and of substitution for the compounds of the aromatic series, founded on the property of salts of manganese, of developing or facilitating oxidation when introduced into an oxidising medium. The oxidation of oxalic acid, by hydrochloric and nitric acids, under the influence of sulphate of manganese, was exhibited.

M. Hanriot presented, on behalf of himself and M. Reynaud, the continuation of their researches on isoxazolones.

M. Le Chatelier found in the residue left, after the action of water on carbide of calcium, besides carbide of silicon, silicide of iron and silicide of calcium.

M. Béal presented a note on behalf of M. Prud'homme, who, in studying the action of a mixture of soda and dilute alcohol on the *p*-nitrodiamidotriphenylmethanes, found that a migration of one of the atoms of oxygen of the NO₂ group was produced, and that there was formed carbinol, or the base of a diamidotriphenylmethane, having the group NO in a position similar to that in the benzenic nucleus, not aminised. His researches also confirm the formula attributed to the fuchsines by M. Rosenstiehl.

Liquefaction of Fluorine.—H. Moissan and J. Dewar.—Already inserted in full.

Notes on the Application of Acetylene to Lighting Purposes.—L. M. Bullier.—These experiments have been carried out on pure acetylene, and on mixtures of acetylene with air, nitrogen, carbonic acid, carbonic oxide, hydrogen, and coal-gas, and they prove that if we cannot generally replace coal-gas by acetylene, in all its applications to lighting purposes, the addition of foreign gases will render its use much simpler, and enable us to obtain results which can now only be obtained with great difficulty, and this without the aid of recuperative apparatus or anything delicate and costly.

On some New Methods of Transforming Paranitrodiamidotriphenylmethanes into Fuchsines, or into Bases of corresponding Fuchsines.—M. Prud'

homme.—The author has tried four methods, viz.:—In an acid medium, with heat; in an acid medium, in the cold; in an alkaline medium; and with sulphide of sodium. He finds the latter method the most advantageous, both as regards quality and quantity; but it does not appear to be possible to obtain more than 60 per cent of the theoretical quantity available: this is attributed to a partial reduction of the base into leucobase. He has demonstrated the existence of two forms of the bases of fuchsines, which is not in contradiction to the observations of M. Rosenstiehl or of H. Weil.

MISCELLANEOUS.

British Association for the Advancement of Science.—The following are the names of the Officers and Committee of Section B (Chemical Science) at the Toronto Meeting of the British Association:—

President—Prof. W. Ramsay, F.R.S.

Vice-Presidents—Prof. G. F. Barker; Prof. F. W. Clarke; Prof. H. B. Dixon, F.R.S.; W. R. Dunstan, F.R.S.; Prof. B. J. Harrington; Prof. E. W. Morley; Prof. W. H. Pike; Prof. I. Remsen; Prof. W. C. Roberts-Austen, F.R.S.

Secretaries—Prof. W. H. Ellis; Arthur Harden (Recorder); Charles A. Kohn; Prof. R. F. Ruttan.

Committee—Prof. W. W. Andrews; Prof. H. E. Armstrong, F.R.S.; Prof. B. Brauner; Prof. C. Le Neve Foster, F.R.S.; Prof. W. L. Goodwin; A. Vernon Harcourt, F.R.S.; O. S. James; F. B. Kenrick; Prof. Loeb; Prof. H. McLeod, F.R.S.; Prof. Meldola, F.R.S.; Prof. Meslans; W. L. Miller; Prof. Nef; H. Ramage; W. W. Randall; Prof. T. W. Richards; F. J. Smale; Prof. A. Smith; A. Stansfield; Prof. W. C. Williams.

The Papers brought before the Section were as follows:—

President's Address—"An Undiscovered Gas."

Prof. W. W. Andrews—Reform in the Teaching of Chemistry.

Report of the Committee on the Teaching of Science in Elementary Schools.

Report of the Committee on Preparing a New Series of Wave-length Tables of the Spectra of the Elements.

Report of the Committee to inquire into the Proximate Chemical Constituents of the various kinds of Coal.

Report of the Committee on the Isomeric Naphthalene Derivatives.

Report of the Committee on the Action of Light on Dyed Colours.

Prof. Ramsay—Helium.

Prof. Brauner—Contributions to the Chemistry of the Rare Earth Metals.

Prof. Brauner—The Atomic Weight of Thorium.

Prof. T. W. Richards—The Atomic Weights of Cobalt and Nickel.

M. Travers—The Occurrence of Hydrogen in Minerals.

Prof. W. N. Hartley and H. Ramage—Spectroscopic Examination of Minerals and Metals.

Prof. Meslans—Demonstration of the Preparation and Properties of Fluorine.

Profs. Moissan and Dewar—The Properties of Liquid Fluorine.

Prof. Ramsay—Demonstration of the Spectra of Helium and Argon.

Dr. J. Waddell—The Permeability of Elements of Low Atomic Weight to the Röntgen Rays.

Dr. J. H. Gladstone and W. Hibbert—Continuation of Experiments on Chemical Constitution and the Absorption of X Rays.

Dr. W. J. Russell—On the Action exerted by Certain Metals on a Photographic Plate.

Prof. H. B. Dixon—Photographs of Explosion Flames.
F. P. Dunnington—Titanic oxide.
F. P. Dunnington—Deliquescence and Efflorescence of Certain Salts.
J. Waddell—Notes on Concentrated Solutions of Lithium and other Salts.
W. L. T. Addison—The Formation of Crystals.
E. C. C. Baly—A Compound of Ozone and Mercury.
J. W. Walker—The Interaction of Hydrobromic and Bromic Acids.
F. T. Skutt—The Composition of Canadian Virgin Soils.
Prof. W. H. Ellis—Analysis of some Pre-carboniferous Coals.
Prof. P. C. Freer—The Constitution of Aliphatic Ketones.
Prof. J. U. Nef—The Chemistry of Methylene.
Dr. A. Lehmann—Formation of a Benzene Ring by Reduction.
Dr. C. A. Kohn—Condensation Products of Aldehyds and Amides.
Dr. Hugh Marshall—A New Form of Bunsen-burner.
Prof. W. C. Roberts-Austen—Molecular Movement in Metals.
Dr. T. K. Rose—The Causes of Loss Incurred in Roasting Gold Ores containing Tellurium.
H. C. Jenkins—The Behaviour of Lead and of some Lead Compounds towards Sulphur Dioxide.
Dr. W. L. Miller and T. R. Rosebrough—The Vapour-Tensions of Liquid Mixtures.
Dr. C. A. Kohn—The Electrolytic Determination of Copper and Iron in Oysters.
Prof. Henri—The Nitro-Alcohols.
Prof. W. W. Andrews—The Plaster of Paris Method in Blowpipe Analysis.
R. Ransford—Some Experiments with Chlorine.
Report of Committees—
 The Electrolytic Methods of Quantitative Analysis.
 Isomeric Naphthalene Derivatives.
 The Direct Formation of Haloids from Pure Materials.
 The Bibliography of Spectroscopy.
 The Carbohydrates of Barley Straw.

Schools of Chemistry, &c.—The following information has been received too late for insertion in the "Students' Number":—

SHEFFIELD TECHNICAL SCHOOL (UNIVERSITY COLLEGE, TECHNICAL DEPARTMENT).—Lecturer in Chemistry, F. Ibbotson, B.Sc., F.C.S.; Professor of Metallurgy, J. O. Arnold; Lecturer, A. McWilliam, A.R.S.M.; Demonstrator and Lecturer on Fuel, F. K. Knowles. The work is divided into two departments:—I. The Technical Department of the University College, including Mechanical, Electrical, Civil, and Mining Engineering; Metallurgy. II. The Evening Department, for providing Technical Instruction for persons engaged during the day in the local industries. The courses of Evening instruction include Magnetism and Electricity, Inorganic Chemistry, Experimental Laboratory, and Electrical Engineering. The Metallurgical Department has been equipped with a view to thoroughly meeting the requirements of the local industries. The Laboratory is fitted with the most modern apparatus for metallurgical analysis, more especially with appliances for the rapid and accurate chemical examination of Iron and Steel, Fuel, and Refractory materials. It also contains a complete pyrometric installation, and a new laboratory for the study of the micrographic analysis of metals has been completed and fully equipped with specially designed microscopes, by Ross, polishing tables, driven by an electric motor, etching appliances, incandescent light for evening work, &c. The School is now the most complete of its kind for teaching the practical manufacture, the chemical constitution, and the physical properties of steel. Special attention is given to the determination of the microscopic

constituents of steel. Although the chief industry of the district occupies the central position in the course of instruction, general metallurgy is not neglected, but is dealt with in a separate syllabus, dealing with metals (other than iron and steel) used in the arts. Students are thus enabled to select and at once enter upon a course of scientific metallurgical training of immediate practical utility. They may take up and work through any portions of the course, but certificates will be granted only to those who follow the prescribed courses and pass the necessary examinations. The course of study is as follows:—Preparatory Chemistry, Metallurgy Lectures, Fuel Lectures, Practical Metallurgy, Practical Fuel Course, Geology and Mineralogy, and Electro-Metallurgy.

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Fig. 11. No. 91.

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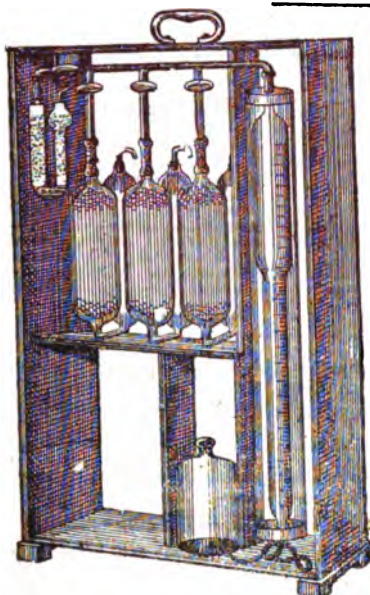
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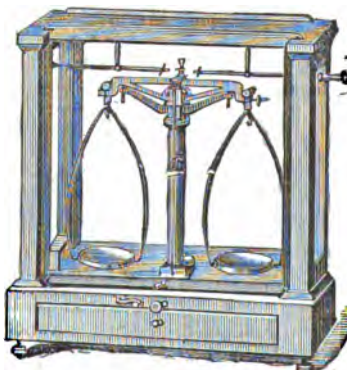


FIG. 3a.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1974.

ON THE PROPERTIES OF NITROBENZENE.

By RICHARD J. FRISWELL.

DURING the severe winter of January, 1886, a drum of nitrobenzene crystallised, and I succeeded in removing from it a large, tabular, transparent crystal, 2½ inches long, 1 inch broad, and ⅝ inch thick, weighing over 15 grms. It was irregular, had no faces, and resembled a mass of ice in appearance. I took advantage of this circumstance to make determinations of the specific gravity of solid nitrobenzene. An Oertling balance was placed in the open air, and allowed to remain until the temperature of an accurate thermometer registered 3·5°, and the crystal was then weighed in air and in water in the usual manner.

Weight in air, 15·849 grms.

Weight in water, 4·059 grms.

Weight of water displaced, 11·790.

Temperature of water, 1·5°.

This gives the sp. gr. as 1·3439, which is correct to ±0·0001.

A large portion of the crystalline mass was then freed from liquid and fused in a paper filter; during the fusion a small quantity of moisture from the air condensed on the melting crystals, but these traces of water were intercepted by the filter, which was rendered waterproof by being saturated with the melted nitrobenzene.

The sp. gr. of the liquid nitrobenzene was ascertained by a very accurate Westphal balance. At 3·8° this was found to be 1·2210, compared with water at 15°, for which temperature the balance is standardised.

During the cooling to 3·8° the liquid became slightly turbid, indicating that the moisture condensed on the fusing crystals had been dissolved to a minute extent.

Correcting the above numbers for water at 4°, we obtain 1·3440 as the sp. gr. of solid nitrobenzene, and 1·2220 as that of the liquid nitrobenzene. The temperature of the liquid nitrobenzene was actually 3·8°, but this I have taken as equal to 4°. I have not attempted to correct the solid nitrobenzene for the increment between 1·5° and 4°, as I have no data for the expansion of the solid substance.

There is thus a contraction of volume amounting to 0·099837, or nearly ⅓, as nitrobenzene passes from the liquid to the solid state, whilst conversely, on fusing, the substance expands from 1·000000 to 1·099804.

Temperature of Solidification of Nitrobenzene.

After the specific gravity of the fused mass of crystals had been taken, a thermometer was immersed in the liquid, and a crystal having been dried on filter-paper was introduced, and the whole stirred. It almost immediately commenced to solidify, and the thermometer rose sharply to 5°, at which it remained constant; a thermometer kept in a large mass of the fusing crystals also registered this temperature.

It has been already stated that the liquid obtained from the fused crystals exhibited a slight opalescence or turbidity, due to the presence of moisture condensed from the atmosphere and dissolved by the nitrobenzene to a minute extent. As soon as the crystal was added, and the temperature of the liquid began to rise, the whole became clear, thus indicating in a striking manner the increased power of dissolving water caused by even so small a thermometric rise as from 3·8° to 5·0°.

Schultz (in *Die Chemie des Steinkohlentheers*, &c., 1882, first edition, p. 355) states, without quoting his authority,

that nitrobenzene has a fusion-point of 3°, a sp. gr. = 1·2002 at 0° and 1·1866 at 4°, no statement as to the temperatures of the water with which comparison is made being given. In the second edition of this work the sp. gr. is stated to be 1·208 at 15°.

Beilstein gives the same numbers, and quotes Mitscherlich as his authority for the fusing-point and Kopp for the specific gravity. Gmelin gives Mitscherlich as authority for both numbers, giving 3° as the fusion-point and 1·209 as the specific gravity. It would appear that no recent observation of these constants has been made.

This year I have repeated the experiments (with the exception of that on the specific gravity of the solid) with a specimen of nitrobenzene prepared from benzene of the highest purity, the nitrobenzene itself being also very carefully purified and twice distilled. The temperature of fusion was obtained with great care by immersing a standard thermometer in a large mass of the solidified nitrobenzene. The thermometer is one made by Casella in 1872; it has a Kew certificate, and has been tested at Kew on two occasions since the date of the original certificate, the last being in 1891. It is a Fahrenheit thermometer, and the correction at 40° to 50° is -2°.

Repeated readings of the former point were taken with this thermometer, and were found to be 43° to 43·1° F.

Several experiments were also made with nitrobenzene obtained by melting well-drained crystals used in the last experiment, cooling below the solidifying point, and introducing the same thermometer, to which some crystals were adhering. The thermometer fell sharply to 41° F., and then the liquid instantly crystallised, the temperature rising as sharply to 43° F., long crystals shooting with extraordinary rapidity through the mass. The number above given, on applying the correction, becomes 41° F. = 5° C.

The specific gravity was 1·2123 at 13° and 1·1974 at 28° compared with water at 15°. Compared with water at 4° these numbers become 1·2116 and 1·1931.

Boiling-point of Nitrobenzene.

Gmelin, quoting Mitscherlich, gives the boiling-point of nitrobenzene as 213°, whilst Schultz (*loc. cit.*, 1882), in his first edition, gives 210°, and in his second (1886) 206° to 207°. Beilstein quotes Städel as giving 205° at 730 m.m.

The sample was boiled on fragments of platinum foil in one of the pear-shaped flasks used for determining the boiling-points of the aromatic hydrocarbons. A Geissler normal thermometer was used. When the boiling had become steady, the readings were 203·4°, 203·4°, 203·5°, 203·5°, 203·5°, and the liquid was distilled to dryness without further rise. The barometer stood at 755 m.m., or 29·73 inches. Correcting for 760 m.m., the boiling-point 203·5° becomes 203·77°.

The boiling-point, when the mercurial column was totally immersed in the boiling vapour, was 209·8°, 209·9°, 209·8°, 209·9°, 210·0°, 209·9°, pressure being 756 m.m. Taking 209·9°, and correcting for barometer at 760 m.m., this becomes 210·2°. The distillation was not continued to dryness, as the previous experiment had proved the liquid to be homogeneous.

The Geissler thermometer had been in my possession for many years; but having noted the fact that the correction for the Fahrenheit thermometer had changed from +0·1° to -2·0° in twenty years, the zero point of the Geissler thermometer was re-determined and found to be +1°. This corresponds with 1·8° F. It is interesting that two thermometers repeatedly used for liquids boiling near 200° C. should exhibit so very similar an alteration in the zero point.

If this correction is applied, the boiling-points become uncorrected for immersion of column 202·8°. With column immersed, 209·2°.

Summary of Results.

The specific gravity of solid nitrobenzene *d* 15°/4° is

n_D^{20} 1.3440, whilst for liquid nitrobenzene it is d_4^{20} 1.2220; d_4^{13} 1.2116; d_4^{28} 1.1931.

Expansion on fusion, 0.099804.

Contraction on solidification, 0.099837.

Boiling-point at 760 m.m., 209° corrected (that is, with column immersed in the vapour).

Nitrobenzene is remarkable as giving a strongly-coloured vapour. The colour, which resembles that of diluted chlorine, is visible in a layer of 2 inches, and is very marked in a layer of 6 or 8 inches. The vapour does not exhibit a banded spectrum when examined by transmitted light.—*Transactions of the Chemical Society*, 1897.

REMARKS ON THE PART PLAYED BY CHEMISTRY IN PERFUMERY.

By LOUIS OLIVIER.

THE fears expressed by M. J. Rouché on the subject of the introduction of chemistry into the perfumery industry suggest a remark that he would certainly not wish me to express. It is only natural that French perfumery, endowed by nature with a kind of monopoly, fears seeing itself robbed of this monopoly, and that through the progress of chemistry. But this evolution is, if I mistake not, a law of Nature. As it spreads through civilisation, science, an international possession, tends to destroy, for the good of the many, the privileges of the few. It is to the multitude of consumers that it holds out its blessings, and thanks to them a larger number of human beings are enabled to enjoy, and it is continually lowering, the selling price.

Whether we rejoice or are sorry, there is the law. Should we therefore on this account, simply with the view of postponing the end of a privilege, turn our backs on this science, in which our foreign rivals see their safety, and which will permit them to beat us in our own products?

To try and avoid an inevitable *evolution*, should the French perfumery industry go on rather to a *revolution*, which would be fatal to it? Instead of letting Germans, English, and Americans make artificial products, of mediocre value perhaps, but of large sale, and thus, to the detriment of the French, take possession of the International market, would it not be better to put oneself in the van of progress, direct it, and assure oneself, independently of the present market, of the acquisition of new ones?

A chemical laboratory and good chemists doubtless represent a serious expense, the more difficult to bear inasmuch as in scientific research there is always the risk of not being immediately successful. But in commerce sacrifices are necessary; we must learn to prepare for the future. If French perfumery decides to have a merry life, it will undoubtedly run the risk of having a short one.—*Revue Générale des Sciences*, 8th year, No. 16, Aug. 30, 1897.

ON THE ESTIMATION OF OXIDE OF IRON AND ALUMINA IN PHOSPHATES.

By N. BLATTNER and J. BRASSEUR.

THE following methods have been studied in the laboratory of the Kuhlmann works at Loos:—

1. The acetic method, or, to be more exact, the acetic acid and ammonia method, carried out in the manner proposed by Maret and Delattre.
2. Glaser's method, based on the preliminary separation of the lime.
3. Lasne's method, by separating the alumina in the

state of phosphate, by means of caustic soda, hypophosphite, and acetate of ammonium.

4. Gruber's method, analogous to the preceding one.
5. Gladding's method, based on the same principle, but making use of caustic potash instead of soda.
6. Thomson's method, by the direct precipitation of phosphate of iron and alumina with ammonia.

MM. Blattner and Brasseur have been led to the following conclusions:—

1. Acetic method. This should be disused: the figures found for alumina are nearly always much too low, the acetic liquid keeping in solution a sensibly constant quantity of alumina.

2. Glaser's method (the alcoholic) gives sufficiently accurate results when the phosphates are free from manganese; it is rapid and easily performed.

3. The caustic soda method gives results scientifically exact, when we observe all the details of the process such as they were described by H. Lasne (*Bull. Soc. Chim.*, xv., p. 118).

4. Gruber's method (described in the *Zeitsch. f. Ang. Chem.*, p. 741, 1896) is nothing but an abridgment or mutilation of Lasne's. It gives inexact results, and should be rejected without hesitation.

5. The caustic potash method, published by Gladding (*Journal of the American Chemical Society*, No. 8, 1896), is analogous to that of Lasne, from which it differs only by modifications in the details of procedure, which might become sources of error.

6. The method of direct precipitation, sufficiently difficult to obtain complete neutralisation (which is indispensable), gives varying results, according to the nature of the phosphate, and the precipitate always contains lime.—*Bull. Soc. Chim.*, Series 3, vol. xvii.-xviii., No. 15.

THE DIRECT SEPARATION AND THE QUANTITATIVE DETERMINATION OF CHLORINE, BROMINE, AND IODINE IN ORGANIC SUBSTANCES.

By P. JANNASCH and E. KOLITZ.

For this purpose the specimen is opened up either by the method of Carius or by combustion with caustic lime.

The method of Carius, especially if the quantity of substance is but small, cannot be carried out as quickly and safely as the decomposition in the lime tube.

The further treatment of the melt is the same as in the following lime method:—

For its execution we use potash glass tubes of 50 c.m. in length and 4 m.m. interior diameter, and charge them as follows:—The tube is first filled by means of a tube-funnel, to the depth of 3 to 4 c.m., with quicklime; then the weighed substance loosely ground up in a tall porcelain mortar is added; the mortar, funnel, and tube are then repeatedly rinsed out with finely-ground quicklime, with which 47 c.m. of the tube are filled. The tube is then closed with a loose plug of asbestos, through which a channel, not too narrow, is formed from end to end by tapping it from side to side.

After the tube has become cool again, the reacting mixture is transferred into a litre flask capable of being closed with a ground stopper, and filled to one-third with water. The tube is then rinsed out, at first with water alone and finally with dilute nitric acid.

With continuous shaking and repeated refrigeration, strong nitric acid is added by portions until there is left only a small residue of undissolved caustic lime, and filtered off from this and the liberated carbon. The caustic soda and residual carbon are well washed out with hot water; the liquid on standing (before filtration) must appear absolutely colourless, and the air in the flask must be inodorous. The filter-paper during filtration must not assume a bluish tint.

The silver-haloid, collected and washed, is introduced whilst still wet along with the paper into a silver crucible, and fused with 5 to 6 grms. of chemically pure soda, so as to form a clear quiet liquid.

When the crucible has become cold the melt is taken up with water, preferably on the water-bath.

The caustic lime resulting was found, in every experiment, perfectly free from halogens.—*Zeit. Anorg. Chemie*, xv., p. 68.

SEPARATION OF CHLORINE AND BROMINE IN PRESENCE OF ACETATES, SULPHATES, AND NITRATES.

By P. JANNASCH and E. KOLITZ.

As regards the direct quantitative separation of chlorine and bromine by permanganate in a strong acetic solution, the attempt was made to effect it in presence of large quantities of sodium acetate. The object in view was to raise the boiling-point by the presence of the acetate, and in this manner to facilitate the liberation of the bromine. But it appeared that this purpose was not only not effected, but that the quantitative liberation of the bromine was actually hindered by the addition of acetates. In a series of such attempted separations only very small quantities of bromine distilled over, whilst the main quantity remained behind. Analogous experiments were made with additions of sodium sulphate and sodium nitrate, with results showing that the presence of each is favourable to the reaction, and does not in any manner interfere with the accuracy of the determination of the two halogens then jointly present.

From the observations of the authors and of Azoff, it may be concluded in general that in presence of alkaline fluids the latter may be neutralised or slightly acidulated with sulphuric or nitric acid, but never with acetic acid.—*Zeit. Anorg. Chemie*, xv., p. 66.

A FLAVOUR-PRODUCING MICROCOCCUS OF BUTTER.

By SIMEON C. KEITH, Jun., S.B.

In April, 1896, I was studying the effects of various bacteria upon cream, and in the course of my experiments I isolated from a mixture of bacteria growing in an agar tube a micrococcus that was found to produce a decided butter flavour and aroma when grown in milk or cream. This proved to be a new species, for which I propose the name *Micrococcus butyri-aromafaciens*.

It has always been the custom to allow cream to sour or "ripen" before churning it for butter, because after this process the butter comes better and more quickly, is of better texture and flavour, and keeps better than butter made from sweet cream. Lord Lister and Pasteur, many years ago, showed that the souring of milk and cream is due to a process of fermentation during which the milk sugar is converted into lactic acid, and that this is due to the activity of minute micro-organisms. It remained for Professor Vilhelm Storch, of Copenhagen, however, to introduce the use of pure cultures of milk-souring bacteria in butter making. Storch found that several kinds of acid-producing bacteria are concerned in the normal souring of cream, and he isolated three species that impart especially fine flavours to butter under favourable conditions.

A similar line of work was taken up by Professor Weigmann, of the Agricultural Experiment Station at Kiel, in Germany, and by Professor H. W. Conn, of Wesleyan University in the United States.

Of the bacteria that have been described as producing a beneficial effect in the ripening of cream, *Micrococcus*

butyri-aromafaciens most nearly resembles Conn's *Bacillus* No. 41 (Storrs Agricultural Experiment Station, *Bulletin* 12, and *Report* for 1894) in its effects upon milk, but it differs in its morphological and in many of its physiological characters. It is a micrococcus growing at 37° and 20° C. It liquefies gelatin slowly, and does not grow well on potato. It may be noted in this connection, however, that recent cultures on gelatin seem to show that the organism has lost to a considerable extent its power to liquefy gelatin during a year's cultivation in the laboratory.

The culture of the micrococcus for use in creameries is propagated in bouillon in Fernbach flasks (broad flasks so constructed that a large surface of liquid is presented to the air). When ready for shipment, the culture is transferred to sterilised bottles under aseptic conditions and hermetically sealed by means of sterilised corks and melted paraffin. Put up in this way, the culture may be kept for an indefinite time without danger of infection by any other organism, but in the sealed bottles the micrococcus loses its vitality so rapidly that after eight days it will no longer produce the best results. Experiments made on a commercial scale show that cream ripened with the aid of fresh, pure cultures of this organism produces generally better butter than the same cream ripened in the usual way. The distinguishing characters of the species are given in the following systematic description.

Micrococcus butyri-aromafaciens, Nov. Sp.

OCCURRENCE.

Isolated from a mixed culture growing on agar in April, 1896.

GENERAL CHARACTERS.

Shape and Arrangement.—A micrococcus occurring generally in pairs.

Size.—0.5 to 0.7 μ in diameter, occasionally reaching 1 μ .

Motility.—Non-motile.

Spore Formation.—No spores.

Relation to Temperature.—Grows rapidly at 37° and 20° C.

Relation to Air.—Aerobic.

Relation to Gelatin.—Slow liquefier.

Colour.—Non-chromogenic (white).

Stain.—Stains well with carbol-fuchsin.

GELATIN.

Slick Culture.—Five days. The gelatin is liquefied in the form of a deep cup $\frac{1}{2}$ inch in diameter. The liquefied gelatin remains clear, with a white film and sediment. The growth below the point of liquefaction is a moderately thick white dotted line.

Plate Culture.

Surface colonies: The colony first appears as a white raised dot which soon sinks in a pit of liquefied gelatin, and ultimately becomes surrounded by a slight whitish ring along edge of the liquefied gelatin.

Submerged colonies: The submerged colonies occur as smooth spherical dots.

AGAR.

Streak Culture.—A very white, smooth, shining growth, which is fairly abundant. The growth is of equal thickness throughout.

Plate Culture.—Characters of no diagnostic value.

Lactose-litmus-agar.—Litmus reddened slightly.

POTATO.

There is very little growth on potato. In two weeks it appears as a very thin white line, barely visible.

MILK.

Not coagulated. A slightly sourish, pleasantly aromatic "buttery" flavour. Slightly acid.

SMITH SOLUTION.

No gas produced. The growth occurs mostly in the open limb of the fermentation tube, the bouillon of the closed limb being only very faintly turbid.

NITRATE.

Reduced to nitrite. Recent cultures do not seem to give this reaction very strongly, although when first isolated it was very marked.

BOUILLON.

Two days, 25° C. Very cloudy with sediment. One week, no further change.

Two days, 37° C. Very cloudy with sediment and ring growth on tube at surface of the liquid.

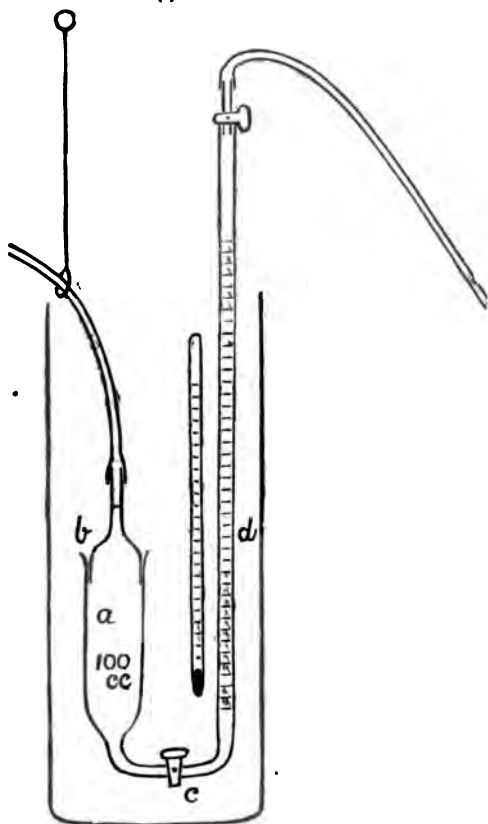
—*Technology Quarterly*, vol. x., No. 2.

SIMPLE LECTURE APPARATUS.

By Prof. W. R. HODGKINSON, Ph.D., F.R.S.E.

VOLUMETER.

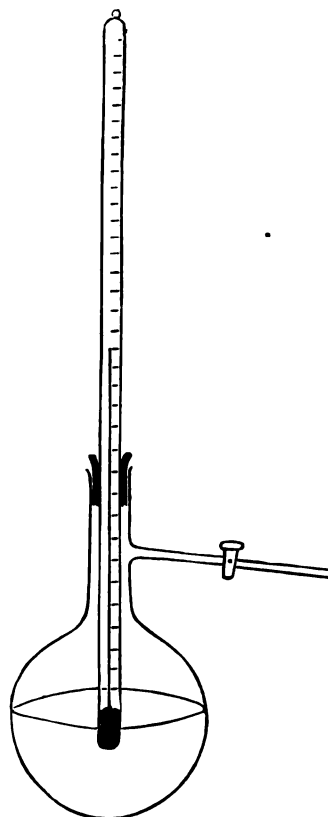
THE apparatus here figured was constructed from the absorption tube of an Orsat-Muencke's gas-analysis apparatus, which had broken at the bend, and an ordinary 50 c.c. burette, the two being joined by sealing a short stopcock tube between (c).



Evidently any convenient liquid may be used for a relative weight or specific gravity determination. Sufficient liquid is introduced to exactly fill the wide tube to the mark *b* and to stand at a moderately low mark on the c.c. tube (*d*), which is read off; the liquid is then lowered from the wide tube to some distance below the stopper, the latter carefully taken out, and allowed to hang by the rubber tube, and the weighed substance (in powder or small pieces) introduced, the stopper replaced, and the liquid carefully brought back to the mark (*b*). The liquid will now stand higher in the graduated tube (*d*); the difference in reading gives the volume in c.c.

The whole apparatus may be sunk in a deep beaker or cylinder of water, which may be retained at any particular temperature during the experiment. The rubber tube attached to the top of the burette serves to apply air pressure to drive the liquid to the mark in the wide tube. This is best done by closing the bottom tap (*c*), blowing strongly into the burette, and closing the top tap. On carefully opening the bottom tap the liquid will rise in the wide tube and the height can be easily adjusted.

The ancient experiment by which is shown that water boils in a closed inverted flask when cold water is poured over it can be somewhat improved upon by employing a distilling or fractionating flask with a stopcock sealed on the side tube and a wide thermometer, wedged by rubber ring in the neck, just dipping into the water. In this case the flask need not be inverted. Other liquids besides water may be used. The temperature can be read off at



which the liquid goes through the process of boiling. The size of flask depends to some extent on the liquid to be employed. For liquids of higher boiling-point than water, a 500 c.c. capacity is enough, and water must not be used for cooling; air from a small bellows is sufficient. For water a 1000 or even 1500 c.c. flask is not too large if the experiment is to be seen at a distance. The mode of working is apparent from the sketch.

Royal Military Academy, Woolwich.

Oxidising Action of α -Monochlorised Camphor.—H. Vittenet. — In the reaction between the aromatic amines and α -monochlorised camphor the authors were surprised to find that, with certain of these bases, colouring-matters were formed which are generally obtained by the direct action of oxidising agents.—*Bull. Soc. Chim. de Paris*, xvii.-xviii., No. 14.

ON THE
PURIFICATION AND ATOMIC WEIGHT
OF CERIUM.*

By MM. WYROUBOFF and VERNEUIL.

(Concluded from p. 139).

Atomic Weight.—Here we come across altogether unexpected difficulties. The sulphate is, up to the present, the only well-crystallised salt of which we can make use, very stable and very easy to purify; now nothing is more difficult than to separate the sulphuric acid from the cerous oxide. Precipitation by oxalate of ammonium (Wolf, Wing) is no good, for oxalate of cerium is not insoluble in acetate of ammonium, and, further, it carries sulphuric acid with it.

The twice-repeated precipitation with soda-lime (Schützenberger) has two drawbacks: if the soda is not in great excess it will not completely decompose the basic sulphate which is formed; in fact, by calcining the oxide of cerium obtained, in a current of hydrogen, we notice very distinctly a more or less considerable disengagement of sulphuretted hydrogen.

If the quantity of soda is sufficient to effect the decomposition, it dissolves cerous oxide, which can be detected by adding a little peroxide of hydrogen. It is true we can separate the cerium and the sulphuric acid integrally in another manner; to do this it suffices to add to the solution an excess of peroxide of hydrogen and caustic soda until the reaction is alkaline, boil to transform the reddish brown peroxide into yellow ceroso-ceric hydroxide. The oxygen thus precipitated and calcined in a current of hydrogen will be found to be perfectly free from sulphur. But this separation does not remove all the difficulties. Modern researches (Ripper, Richards and Parker, &c.) have shown, in the most distinct manner, that the estimation of sulphuric acid in sulphates, in the presence of chlorides, can only be done with a very insufficient approximation, for an operation so delicate as the determination of an atomic weight. We could certainly make the correction for the chlorine entangled by the sulphate of baryta, but Messrs. Parker and Richards have shown that this correction does not always lead to a better result, because of the solubility of barytic sulphate in an acid solution, and further, in the present case we do not know in what condition the entangled chlorine is present. Is it in the state of BaCl_2 or NaCl , or both together? An example will show what variations in the atomic weight can occur according to the method adopted of making the correction. We had in the sulphate of the first fraction of Series I. (see Table), for 100 parts of anhydrous cerous sulphate, 123.384 of SO_4Ba , which gives us the atomic weight—

$$\text{Ce} = 93.01.$$

This sulphate of baryta contained 0.27 per cent of Cl ,† which, counted as NaCl , gives 122.838 of SO_4Ba and an atomic weight of 93.84; counted as BaCl_2 , it gives 122.401 of SO_4Ba and an atomic weight of 94.53. The true atomic weight, as we shall prove further on, is very close to 92.7. We see thus that the method of estimating the sulphuric acid cannot be applied to the determination of the atomic weight of cerium.

M. Brauner made use of another method which at first sight appeared to be quite beyond reproach. He strongly heated the dehydrated sulphate, and calculated the atomic weight from the weight of Ce_2O_3 obtained. The average of his experiments, which were very closely concordant, gave—

$$\text{Ce} = 93.48.$$

Schützenberger however remarked, with very good reason,

that the ceroso-ceric oxide had different weights according to the temperature of calcination, and that therefore one could not tell with accuracy the moment when its composition was really Ce_2O_3 . These variations may attain several thousandths, so he considered that the process could not furnish really exact results.

We can only recall from memory the determinations made by starting with cerous oxalate (Jegel, Bührig, Rammelsberg). We consider this salt as altogether unfit for deciding the atomic weight of cerium; firstly, because it is almost impossible to obtain it in a state of purity, for when it is crystallised it always retains cerous oxalate; and secondly, because organic analyses are subject to errors far too great to allow of their application to so high an atomic weight. The great differences found (Jegel, 91.66; Bührig, 94.4; Rammelsberg, 92.16) show the inefficiency of the method. It is necessary to remark further—these surprises are not rare in the history of cerium—that Bührig's figure 94.4, generally accepted at the present time, is the same as that given by Marignac in 1848, and shortly afterwards, in 1853, admitted by him to be incorrect. He obtained it, as a matter of fact, by the direct precipitation of the sulphate by chloride of barium. Finally, to complete the history, we will cite the analysis of the anhydrous chloride made by Mr. Robinson, which gave—

$$\text{Ce} = 93.5,$$

a figure identical with that obtained by M. Brauner. The chloride is too deliquescent a salt, and too difficult to free from the oxychloride which accompanies it, to allow of its being used for the determination of the atomic weight with any chance of success. All the old methods being rejected as insufficient, there remains now to find a better. We noticed, while making a large number of estimations in water in the hydrated sulphate, that the figures obtained were very closely concordant. On twenty estimations none varied more than 0.03 per cent. Thus the idea occurred to us to employ this estimation for the determination of the atomic weight; in any case it would serve to fix, in an exact manner, the conditions under which the ceroso-ceric oxide, obtained by calcination at a high temperature, would have the formula Ce_2O_3 , and to check with certainty Brauner's method. The table, in which we give a *résumé* of our experiments, shows in a decisive manner:—

1st. That the estimation of the water gives less variations than the ceroso-ceric oxide calcined at a white heat, and consequently a more exact figure for the atomic weight.

2nd. That Brauner's method gives a fairly exact figure when the sulphate is calcined at a very high temperature (about 1500°), because it is only at this high temperature that we can eliminate the last traces of sulphuric acid. In this state, if it is perfectly pure, it should be perfectly white, without the least trace of any rose, chamois, or yellow tint. By this method it is the lowest figures which are nearest the truth.

But to be able to estimate the water accurately, it is of importance which of the numerous hydrated cerous sulphates is used. We therefore commenced by carefully studying the conditions of the formation, and the properties, of these salts, about which there is great want of accord.

The hydrates described are five in number:—

A.	$(\text{SO}_4\text{Ce})^*$		5 aq.
B.	SO_4Ce		2 aq.
C.	$(\text{SO}_4\text{Ce})^*$		8 aq.
D.	SO_4Ce		3 aq.
E.	SO_4Ce		4 aq.

The crystalline form and the optical properties of the salts A, C, and D have been determined, at least approximately, by Marignac and Des Cloizeaux. The salt E occurs in an efflorescent form, consisting of a tangled mass of needles quite indeterminable; the salt B only

* *Bull. Soc. Chim.*, Series 3, vol. xvii.-xviii., No. 14.

† This figure is only slightly different to the mean of seventeen analyses made by Messrs. Richards and Parker, who found 0.237 per cent (*Zeit. Anal. Chem.*, viii., p. 417).

TABLE (O=16; S=32).

	Hydrated salt.	Anhydrous salt.	Ce ₂ O ₃ .	H ₂ O p.c.	Ce ₂ O ₃ p.c. from Hydrated salt.	Ce ₂ O ₃ p.c. from Anhydrous salt.	Atomic weight calculated from—			
							H ₂ O.	Ce ₂ O ₃ from the Hydr. salt.	Ce ₂ O ₃ from the Anhydr. salt.	
I.	1.	1'2385	0'9875	0'5977	20'267	48'259	60'526	92'84	93'08	93'16
	2.	1'2730	1'0148	0'6138	20'282	48'216	60'484	92'65	92'88	92'95
	3.	1'2030	0'9590	0'5794	20'282	48'162	60'417	92'65	92'64	92'63
	4.	1'5420	1'2295	0'7430	20'265	48'184	60'431	92'85	92'74	92'70
			Averages	..	20'274	48'205	60'464	92'74	92'84	92'86
II.	1.	0'9642	0'7685	0'4642	20'296	48'143	60'403	92'49	92'55	92'56
	2.	1'3260	1'0571	0'6389	20'279	48'182	60'438	92'69	92'72	92'73
	3.	1'1429	0'9112	0'5518	20'273	48'280	60'557	92'76	93'17	93'30
	4.	0'9072	0'7232	0'4372	20'282	48'192	60'453	92'66	92'77	92'80
			Averages	..	20'282	48'199	60'463	92'65	92'80	92'85
III.	1.	1'2114	0'9658	0'5840	20'274	48'208	60'468	92'75	92'84	92'87
	2.	1'2411	0'9894	0'5984	20'280	48'215	60'481	92'68	92'88	92'93
			Averages	..	20'277	48'211	60'474	92'71	92'86	92'90
Average of the three series				20'278	48'205	60'467	92'70	92'83	92'87	
Difference between maxima and minima				0'031	0'137	0'154	0'36	0'62	0'74	
Diff. between maxima and the general average				0'018	0'075	0'090	0'21	0'34	0'43	

has been vaguely described as crystallising in needles similar to those of the salt A. In reality this salt does not exist, and every time we obtain quantities of water of about 16 per cent we are sure of finding under the microscope two sorts of crystals; prisms of the salt A and octahedra much less bi-refrangible of the salt C.

The conditions under which these diverse hydrates are formed are very different, according to whether the liquid does or does not contain a little free sulphuric acid. We have only here to examine the particular case of solutions absolutely exempt from free acid. This condition is essential, for we know that the cerous sulphate—no matter how well it may be crystallised—retains sulphuric acid with a remarkable tenacity, which may be explained perhaps by the facility with which an acid sulphate, described by one of us some time since (*Bull. Soc. Chim.*, Series 3, vol. ii., p. 745, 1889), is formed, and which is only difficultly decomposed at a relatively very high temperature.

To remove the last traces of sulphuric acid we can proceed in several different ways. We can either precipitate two or three times with alcohol, or, after a preliminary dehydration carried out at as high a temperature as possible (400° to 450°), dissolve, crystallise the greater part at 75° to 80°, evaporate the mother-liquor which retains the greater part of the existing free acid, heat until all sulphuric acid vapours are driven off, dehydrate the crystals, dissolve the whole in water, and repeat the operation three or four times; or again, and in a more simple manner, decompose the nitrates with an insufficient quantity of sulphuric acid, and heat to 500°. The mass contains either ceroso-ceric oxide, or a basic salt which remains on the filter, and cerous sulphate which dissolves. The salt thus prepared gives, at all temperatures up to about 85°, only the hydrate C, sometimes mixed—when the temperature does not go beyond 45°—with a few needle-like crystals, generally isolated and sharply defined, of the hydrate D.* It is only at temperatures of about 100° that we obtain clino-rhombic crystals of the salt A always mixed with the hydrate C. On removal from the mother-liquor these crystals effloresce, or rather appear to effloresce very rapidly; in reality they become covered

with a layer of crystals of the higher hydrate C, which is only normal and stable under these conditions.

It follows from this that the salt (SO₄Ce)₃, 8aq., is the only one which can be used for exact weighings. It keeps very well in the air, easily forms limpid crystals, especially when small. Its density at 17° is 2'885. To obtain it quickly in very limpid crystals it is necessary to dissolve the sulphate, dehydrated at about 400°, in ten times its weight of water, and crystallise at 60°. In twenty-four hours we get an abundant crop of crystals, not more than 2 m.m. in diameter, but perfectly transparent. These are drained on filter-paper until quite dry; they are then powdered and dried anew. In this state they are ready for analysis, and give constant results, even after having been exposed to the air for two or three days. The hydrate C, like all the others, is easily dehydrated at about 250°; if then heated to redness in a tube it will not give a trace of moisture. Once dehydrated, it will stand a temperature of 500° without undergoing the slightest decomposition. On these two points we are in complete discord with M. Brauner, who maintains that the water cannot be entirely driven off even at 400°, and that a temperature of 500° turns the salt yellow by oxidation commencing to take place. We would remark that if our estimation of the water was too low, the atomic weight of cerium—already lower than that of M. Brauner—would be still further reduced; on the other hand, the oxidation of cerous sulphate at 500° clearly shows, as Nilson first observed (*Ann. Chim. Phys.*, Series 5, vol. xxx., p. 431, 1883), the presence of thorium.

If the dehydrated salt is heated to about 1500° it only loses its acid very slowly, and it requires to be kept at this high temperature for at least fifteen minutes before its weight remains constant. The oxide thus obtained appears to be free from sulphur, at least that in the analysis I. 2 (see Table), calcined in hydrogen, did not show any trace. However, the extreme difficulty with which this salt parts with its acid by calcination ought to make us consider this method as less exact than that by estimating the water.

To obtain fairly correct results it is necessary to do the calcination in double crucibles, using platinum crucibles as small as possible, not taking more than 1·5 grms. of material, and weighing in an atmosphere free from moisture.

The three series of analyses given in the table were performed on three products, essentially different:—

* These crystals are at once distinguished under the microscope from the needles of the hydrate A, with which they might be confounded. They belong to the hexagonal system, and in polarised light disappear in the direction of their length; the extinction of the crystals A is, on the contrary, very oblique on the face *m m*.

I. Cerium obtained from rough oxalates from monazite, by the process we have described above, and carefully purified from thorium.—This cerium was transformed into sulphate, and the sulphate fractionated in nine portions. The analyses 1 and 2 were done on the first portion, the analyses 3 and 4 on the last.

II. Cerium obtained from the industrial treatment of oxalates extracted from monazite, very rich in thorina (about 50 per cent) by carbonate of ammonium.—This reagent dissolves a certain quantity of cerium, the didymium, and all the yttria earths. After eliminating the great part of the thorina by the usual methods, the remaining mixture was treated the same as the cerium I. The sulphate gave three fractions. The analyses 1 and 2 are of the first, the analyses 3 and 4 of the last.

III. Cerium extracted from rough oxalates obtained from cerite, and purified like I. and II.—The sulphate was separated by three crystallisations at 60°. The analysis 1 is of the first, the analysis 2 of the last.

The examination of this table shows in the clearest manner that cerium, no matter what its origin, properly freed from impurities and especially from thorium, is an element, pure and simple, giving always a perfectly white oxide, Ce_2O_3 , at a high temperature. The indirect methods which have been used do not allow of its atomic weight being definitely determined, but we can say with certainty that it is close to 92.7, with an approximation of 0.2 to 0.3 per cent, rather less than more.

Having now proved the identity of this element, we propose to study its principal combinations, more especially its numerous oxides.

THE ATOMIC MASS OF TUNGSTEN.*

By WILLETT LEPLEY HARDIN.

(Continued from p. 39).

Preparation of Tungsten Trioxide.

THE material used in the first few series of determinations was obtained from wolframite from Zinnwald, Bohemia. The greenish yellow oxide obtained by digesting this mineral for several days with aqua regia was washed with distilled water and afterwards dissolved in ammonium hydroxide. The solution was evaporated to crystallisation, and the ammonium tungstate which separated out was strongly ignited. The resulting oxide was again dissolved in ammonium hydroxide, the solution was evaporated to crystallisation, and the resulting ammonium tungstate strongly ignited. The oxide thus obtained was placed in a porcelain boat in a combustion tube and gently heated in a current of hydrochloric acid gas to remove the last traces of molybdenum. The material was then re-ignited and placed in a large porcelain dish filled with distilled water. Ammonia gas was conducted into the water for several days, after which the supernatant liquid was syphoned off and evaporated to crystallisation. The ammonium tungstate which separated out was ignited and the process repeated. The material obtained from the second crystallisation was used in the first series of experiments.

Reduction Series.

Tungsten trioxide obtained by the method just described was used in these experiments. The reductions were made in a hard glass combustion-tube in a current of hydrogen, which was first conducted through solutions of ammoniacal silver nitrate, potassium permanganate, alkaline lead nitrate, caustic potash, and finally through sulphuric acid and a tube containing anhydrous calcium chloride. The reduction in each case was continued for several hours at a temperature almost high enough to melt the glass tube. The porcelain boat which contained

the oxide was protected from the glass tube by means of platinum foil. The weighings were made on a Troemner short-armed balance with a set of weights which had been previously calibrated. The balance is sensitive to the fortieth of a m.grm. The results, calculated on the basis of $\text{O} = 16$, are as follows:—

	Weight of WO_3 . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	1.64084	1.30100	184.05
2	1.79728	1.42550	184.044
3	2.60739	2.06788	183.98
4	4.57390	3.62890	184.33

At this point an unglazed porcelain tube was substituted for the glass tube, and the reductions that follow were continued for three hours at the highest temperature obtainable in a combustion furnace.

	Weight of WO_3 . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	3.32320	2.63547	183.94
2	6.11056	4.84580	183.91
3	9.23802	7.32393	183.66

The last experiment was continued through a period of eight hours.

The first three results agree very closely and give 184.02 as a mean for the atomic mass of tungsten. The mean of the results with the glass tube is 184.10. The maximum deviation is 0.35. The mean of the results obtained with the porcelain tube is 183.84, with a maximum difference of 0.28. The maximum deviation in the whole series is 0.67.

Oxidation Series.

The metal obtained in the foregoing reductions was used in these experiments. The oxidations were made in porcelain crucibles. The material was protected from particles of dust by means of a porcelain dish suspended a short distance above the crucible. The oxidation in each case was continued until there was no further increase in weight.

	Weight of W. Grms.	Weight of WO_3 . Grms.	Atomic mass of tungsten.
1	1.70220	2.14400	184.94
2	1.37651	1.73393	184.86
3	2.05606	2.58951	185.00
4	1.10300	1.38933	184.91
5	1.85855	2.34143	184.75
6	7.28774	9.18730	184.15

The mean of the first five results of this series is 184.89. This value is almost identical with that obtained by Pennington and Smith. The mean of the whole series is 184.77. The maximum deviation is 0.85.

Reduction of Oxide obtained by the Ignition of Metal.

Inasmuch as the value obtained in the oxidation series is almost a unit greater than that obtained by reduction, it was thought advisable to make a series of reductions of the oxide obtained in the series of oxidations.

	Weight of WO_3 . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	2.02890	1.61071	184.88
2	2.15894	1.71388	184.85
3	2.35206	1.86740	184.94
4	1.39137	1.10351	184.01
5	1.92125	1.52487	184.66
6	1.46746	1.16383	183.99
7	5.01313	3.97560	183.93
8	6.11056	4.84580	183.91

The first three results of this series agree very closely and give 184.89 as a mean for the atomic mass of tungsten. The last three results are equally concordant and give 183.94 as the mean value. The mean of the whole series is 184.40. The maximum deviation is 1.03. The oxide used in these experiments was very light and fluffy.

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xix., No. 8.

The material used in the last experiment was moistened and re-ignited to render it more compact.

Oxidation of Metal obtained from the Second Reduction.

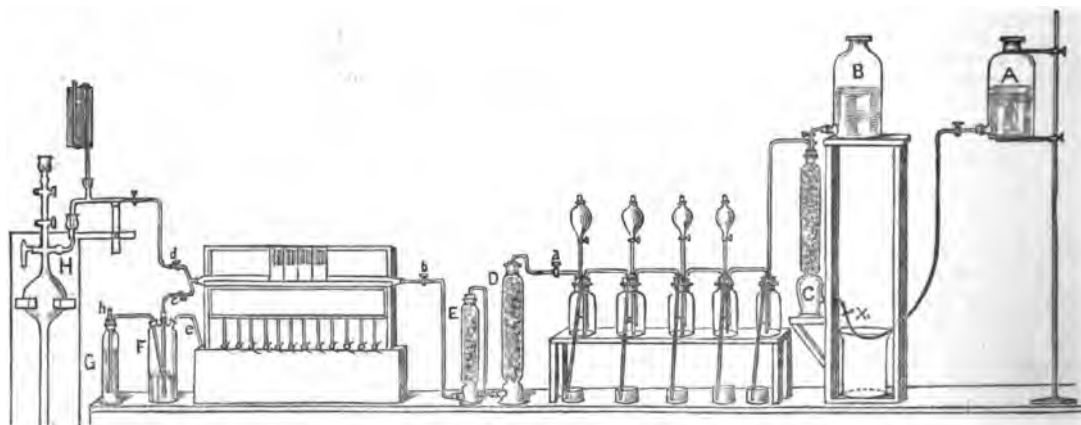
The material obtained in the last series was used in these experiments.

	Weight of W. Grms.	Weight of WO ₃ . Grms.	Atomic mass of tungsten.
1	3.96360	4.99460	184.53
2	2.63034	3.31647	184.01
3	1.60964	2.02804	184.65

The variations in this series are similar in every respect to those of the preceding series.

In view of the wide variations in all the preceding work, an attempt was made to weigh the water formed in the reduction of tungsten trioxide. The moisture was collected in a small glass-stoppered U-tube filled with anhydrous calcium chloride. To this tube was attached another similar tube to prevent absorption of moisture from the air. Several determinations were made, but the results were too discordant to establish anything. The removal of the last traces of air from the generator and wash-bottles was very difficult to accomplish, and hence the results were usually too high. The presence of air, even in small quantities, would also affect the results in

The stopcocks of the different separatory funnels were then opened and the solutions allowed to pass into the corresponding wash-bottles. The first bottle contained pure water, the second ammoniacal silver nitrate, the third and fourth potassium permanganate, and the fifth alkaline lead nitrate. The drying tower *b* was filled with anhydrous calcium chloride and caustic potash, and the tower *c* with alternate layers of glass-wool and phosphorus pentoxide. These were substituted for sulphuric acid, for, according to Dittmar and Henderson (*Proc. Phil. Soc.*, Glasgow), hydrogen when passed through sulphuric acid becomes contaminated, owing to the reduction of the acid by the hydrogen. From the drying-towers the hydrogen passed into a thin-walled glazed porcelain tube, placed in a combustion furnace. The bottles *g* and *f* acted as a regulator; the outlet to *f* was connected to a suction tube, *e*. When the suction was greater than the backward pressure of the wash-bottles, air passed in at *k* and through the columns of sulphuric acid in *g* and *f*. The length of these two columns of acid were adjusted so that the pressure exerted against the air passing through them was equal to the backward pressure of the wash-bottles. The Sprengel pump, *h*, was attached so that the metal might be cooled in a vacuum and thus prevent any occlusion of hydrogen. When the reductions were complete the stopcocks at *b* and *c* were closed. The reservoir



the reduction series. To overcome this difficulty, a different form of apparatus was constructed, the plan of which is shown in the accompanying sketch.

At the beginning of the operation, water, which had recently boiled, was allowed to pass from the bottle, *A*, into the tower, *c*, containing granulated zinc. When *c* was completely filled, the water passed through the small outlet tube into the first wash-bottle; after filling this it passed into the second, and so on. When the water reached the bottom of the cork in the last wash-bottle, the stop-cock at *a* was closed. The stopcock in each of the separatory funnels was then opened and the water allowed to rise until the stems of the funnels were completely filled. In this way the air in the generator and wash-bottles was completely displaced by water. The stopcock at the bottle *A* was now closed and sulphuric acid allowed to drop from the bottle *B* upon the zinc in *c*, the clip at *x* being opened at the same time. The hydrogen formed by the sulphuric acid and zinc forced the water out of *c* into the beaker below, after which the clip at *x* was closed and the clip on the syphon of the first wash-bottle opened. When the water was removed from this bottle, the clip was closed and that of the second syphon opened, and so on until the water in all these bottles was displaced by hydrogen. The separatory funnels were then filled with the different solutions used in purifying the hydrogen. The stopcocks at *a*, *b*, and *c* were opened, while that at *d* was closed; this allowed the hydrogen to pass through the apparatus.

of the vacuum pump was exhausted and the stopcock at *d* opened. This was repeated several times, until the vacuum was almost perfect.

This form of apparatus was used in all the reductions which followed. The air could be completely removed from the apparatus in a short time. The reductions were continued for a period of three hours at the highest temperature obtainable in a combustion furnace. When the quantity of material exceeded 3 grms. the time was longer.

Reduction Series.

All the material resulting from the preceding experiments was ignited and digested for several days with pure aqueous ammonia. A residue was left which gave the bead test for silica. It is evident from this, that tungstic acid, when reduced in a porcelain boat, takes up silica. The solution of ammonium tungstate was syphoned off and evaporated to crystallisation. The crystals of ammonium tungstate were strongly ignited, and the resulting oxide used in the experiments. For the first time the metal was allowed to cool in a vacuum.

	Weight of WO ₃ . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	3.55192	2.81560	183.55
2	4.59362	3.64461	184.34
3	4.30435	3.41459	184.21
4	2.64671	2.09900	183.95

The mean of these four results is 184.01. The maximum difference is 0.79.

Oxidation Series.

The metal obtained in the preceding series of results was re-oxidised, and the following values obtained for the atomic mass of tungsten:—

	Weight of W. Grms.	Weight of WO ₃ . Grms.	Atomic mass of tungsten.
1	2.80958	3.54370	183.70
2	3.63095	4.57662	184.30
3	2.09740	2.64455	183.99

This series, like the preceding, is of little value, owing to the wide variation in the results.

Inasmuch as the removal of air from the present form of apparatus was a matter of little difficulty, another attempt was made to collect the water formed in the reduction of tungsten trioxide, and from its weight calculate the atomic mass of tungsten. The moisture was collected in a glass-stoppered U-tube filled with alternate layers of glass-wool and phosphorus pentoxide. From a series of blank experiments, it seemed that any error introduced by the presence of air in the apparatus would be almost inappreciable. The following results were obtained:—

	Weight of WO ₃ . Grms.	Weight of H ₂ O. Grms.	Atomic mass of tungsten.
1	5.01313	1.16742	184.07
2	2.02890	0.47090	184.86
3	7.04192	1.63864	184.27
4	3.34204	0.77832	184.07

The variations in this series are similar in every respect to those of the different reduction and oxidation series.

(To be continued).

NOTICES OF BOOKS.

The Stamp Milling of Gold Ores. By T. A. RICKARD, M.E., F.R.G.S., A.R.S.M. New York and London: The Scientific Publishing Co. 1897. Pp. 260.

PROBABLY the most important point in gold-winning, and one that in most cases is entirely neglected, is deciding on the best and most economical method of extracting the gold from the ore, after it is brought to bank. Hundreds of thousands of pounds have been lost and wasted, through the ignorance of those in charge of the mine, on this vital question.

A new gold-field is discovered; there is a rush; mushroom experts spring up, who give reports on properties (50 to 500 guineas, according to the proposed capital); companies are floated, operations commenced, and when a certain quantity of ore is being raised the Directors (who as a rule know nothing whatever about the subject) decide to erect a mill; they do so, and very good mills they are, but in ninety cases out of a hundred they are quite unfit for the work for which they have been bought, and, though assays show a payable amount of gold in the quartz, the mill cannot extract it, and about 60 per cent goes away in the tailings. Now this enormous loss could be avoided if only the Directors would consult a known practical man, with a reputation to keep, who could advise them whether their particular grade of ore was more adapted to fast or slow stamping, long or short drop, deep or shallow boxes, inside or outside plates, and all those other *little* details of construction and working the neglect of which leads to 60 per cent loss (a very common figure in the Transvaal).

All these points, and many others, are clearly and thoroughly discussed in these pages.

In Chapter I., the Philosophy of the Stamp-milling Process, the author gives examples of two methods of milling very wide apart in their details, but each well adapted to the class of ore to be dealt with. In Colorado the ore is of complex pyritic character, containing 15 per cent of sulphides, and the gold is in a very fine state of

division; it is therefore necessary to have a long 20-inch drop of about thirty to the minute, a deep box of about 14 inches discharge, and inside plates; on the other hand, in California, where there is only about 1 per cent of sulphides present in the ore, this method would be utterly inadequate; so in that district they use a 5- or 6-inch drop of 100 or 105 per minute, a shallow box, coarser screens, and outside plates only: both these methods, which are now thoroughly understood, sprang from a common origin between the two extremes, and were taken west by the miners from Georgia, but experience and observation has modified them in the manner indicated, to meet the necessities of the ore dealt with.

In succeeding chapters the author describes the methods of milling adopted, after long and costly experience, in all parts of the world:—the early Australian methods, as used at the Clunes, Port Philip, and Colonial Mining Companies; the more modern Australian methods, used at Ballarat, Star in the East, Britannia United, &c.; the Stamp Mills of Otago, New Zealand; and in Chap. XIII. we come to a Review of Australian Processes. In this chapter the relative merits of wet *versus* dry processes are discussed, and Mr. Rickard seems to be of the opinion that the adoption of one or the other is largely dependent on the proximity of the gold-fields to the railway system. In America, as the railway extends, there is a tendency for the smelter to replace the millman, for fire reduction processes to supplant wet methods of gold extraction. In Australia, as yet, the smelter is only to be found in the silver regions; elsewhere the stamp-mill is supreme.

Chapters on Mills and Millmen, and the Future of the Stamp-mill, will be found very interesting reading, and they bring to a close an excellent practical work by a man who shows throughout that he is a thorough master of his subject, and, from a practical point of view, we can only add that we wish there were many more like him.

Bulletin of the Agricultural Experiment Station, Baton Rouge. By D. N. BARROW, Assistant-Director. Issued by the Bureau of Agriculture and Immigration. Series 2, No. 47. 1897.

THE experiments described in this Bulletin are on Corn, Cotton, Forage Crops, and Tobacco. The dry weather has materially cut down the yields, by depriving the crops of the moisture necessary for taking up the available plant food, with the result that all the results have been completely vitiated from an experimental standpoint. Still the yields of cotton and corn were very fair, showing the advantage to be gained by a thorough preparation of the soil and frequent cultivation.

The experiments with corn were a repetition of those made last year. The yields afforded by twenty different varieties are here given, in connection with fertiliser tests. The highest yield per acre was of Farmer's Pride, viz., 35.3 bushels; the lowest being Improved Leeming, which only gave 13.5 bushels per acre. The fertiliser tests with corn are still uncertain and unsatisfactory, owing to the continued recurrence of dry weather for the past eight years just at the time when moisture was most needed. Phosphoric acid in its various forms has always been seen to be beneficial, but there is no definite evidence in favour of one form or another.

The work on the station with regard to cotton has, since the introduction of tobacco, been confined to variety tests, and the results are given in a table. The short staples were ginned and the percentage of lint to seed estimated; the long staples are awaiting the arrival of a roller gin from Europe.

Twelve varieties of forage plants were experimented on, the best results being obtained from *Desmodium mollis*, which yielded 42,429 lbs. per acre; the lowest was of *Teosinte*, which gave only 6120 lbs. per acre.

The experiments with varieties of tobacco were carried out on Plot No. 12, which was formerly devoted to nitrogen experiments on cotton. The rows were 3½ feet wide,

and ran at right angles to the old nitrogen rows. Twenty-two varieties were set, but they all suffered so severely from the dry weather that the results are practically worthless—hence the tabulated results convey no information of any value. In the experiments on fertilisers and tobacco it is singular that, though emphatically a "potash-loving" plant, there is no indication of that substance having benefitted tobacco on this soil.

Since the publication of No. 41 Bulletin the tobacco raised on the experimental station has been worked up into cigars. The cigars were recently on exhibition at the State Agricultural Society, and were pronounced by experts to be of excellent quality, and it is expected that they will soon (in eighteen months or two years) compare favourably with Havanas.

Catalogue of Standard Second-hand and New Books, English and Foreign. On Chemistry and the Allied Sciences, Technology, Optical and Electrical Science, Metallurgy, Mineralogy, Brewing, Dyeing, Manufactures, Agriculture, &c. By WILLIAM F. CLAY. University Book Warehouse, 18, Teviot Place, Edinburgh.

THIS Catalogue (No. 80, 1897), in addition to its usual features, contains an account of works on the constants employed in the analysis of fats and oils. There is also a list of monographs and other original memoirs on chemistry not at present to be obtained in any other form. We feel therefore free to call the special attention of students, and also of librarians, &c., to this list.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 8, August 23, 1897.

Critical Constants of certain Gases.—A. Leduc and P. Sacerdote.—The critical temperatures are obtained by us at less than about 0.5, and the critical pressures at less than 1 atmosphere.

The Absorption of the X Rays.—Abel Buguet.—To determine the relations existing between the thickness of a body and its opacity for the X rays the author uses scales of thickness.

No. 9, August 30, 1897.

Photography of the Fluoroscopic Image.—Charles Porchier.—To oppose an absolute barrier to the X rays I arrange the experiment as follows:—The door of the dark chamber of the laboratory is perforated with an aperture in which is placed the object-lens of my photographic apparatus. Besides a barrier of lead behind the door and all around the object-glass, in a radius of 0.50 metre, I have nailed a sheet of lead of 0.003 metre in thickness. Thus a barrier of lead, the metal screen, and the object-glass protects the gelatino-bromide film absolutely against the action of the X rays. The fluoroscopic image is the most interfered with, and can act alone upon the plate. The screen is placed at 0.57 metre from the objective, and the focus of the phial is only 0.03 behind it.

Bulletin de la Société Chimique de Paris.

Series 3, Vol. xvii.-xviii., No. 14.

M. Ponsot made some observations relative to the congelation of milk; in his opinion it is not proved that cryoscopy alone can prove the addition of water or determine its extent.

M. Delépine gave the results of his thermo-chemical experiments on formic aldehyd; his conclusions as to its molecular formation conform with those of Tollens and Grossmann.

M. Causse described his experiments on the action of

salicylic aldehyd on urea; when a mixture of these substances is heated to 110° they combine with elimination of water, forming salicyl-triurea.

M. Hébert presented, on behalf of M. Rabaut, his researches made on the combinations of cupreous chloride with nitrites.

A Method of Oxidation and Chloridation.—A. Villiers.—Already noticed.

Destruction of Organic Matters in Toxicology.—A. Villiers.—Already noticed.

Products of Decomposition of Carbide of Calcium by Water.—E. Chuard.—Already noticed.

Purification and Atomic Weight of Cerium.—MM. Wyruboff and Verneuil.—(See pp. 137 and 153).

On Saccharines, New Colouring-matters derived from Benzoic Sulphimide (Saccharine).—P. Monnet and J. Koetschet.—A long paper, not suitable for abstraction.

Isomerism existing between Pilocarpidine and Pilocarpine.—A. Petit and M. Polonovski.—From their recently-published researches on these bodies, the authors have been led to doubt the accuracy of the formulae they had adopted, and to suspect the existence of isomerism between these two bodies. The experiments tried confirmed their belief.

Alkalimetric Estimation of Metals: Mercury.—H. Lescœur.—Not suitable for abstraction.

MISCELLANEOUS.

South West London Polytechnic Institute.—Principal, Professor Herbert Tomlinson, B.A., F.R.S. Professor of Chemistry, J. B. Coleman, A.R.C.S., F.I.C., F.C.S. This Institute was opened in 1895, and is therefore commencing its third session's work. Two and three year courses of instruction are given in Chemistry and in Mechanical and Electrical Engineering, suited to the requirements of analysts, industrial chemists, engineers, and others. The chemical department contains two laboratories, balance, lecture, and store rooms, and is designed and equipped for advanced chemical work. In addition to the day courses, evening instruction is given in Pure Chemistry, Pharmacy, Photography, Colours, and other branches of Applied Chemistry.

City and Guilds of London Institute.—A special course of instruction in Electro-chemistry will be given during the coming session at the City and Guilds Central Technical College. The course will include practical instruction in electro-deposition, the use of the electric furnace, dynamos, transformers, and accumulators. A great part of the time of the students attending the course will be devoted to Electro-chemical Research and the study of Electro-chemical Action. Candidates for admission will be required to submit evidence of having a general knowledge of physics and chemistry, and of having been specially trained in one of these subjects.

On the Essence of Bitter Fennel.—E. Tardy.—The French essence of bitter fennel was first washed with an aqueous solution of potash, then with distilled water. The washings, treated with hydrochloric acid, gave anisic acid. The washed essence was then treated with bisulphite of soda, when an abundant precipitate was produced. This precipitate, after being thoroughly washed with ether, was decomposed by potash. The oily liquid produced was submitted to fractional precipitation, by which means it was divided into seven portions, the first boiling below 240° and the latter between 265° and 270°. Above this point a product is obtained of a fatty appearance, in which are a large number of crystalline flakes: these can be separated by dissolving them in ether and re-crystallising. This body combines neither with acids nor alkalies, and on analysis gives the formula $C_{13}H_{14}O_2$.—*Bull. Soc. Chim. de Paris*, No. 13, 1897.

Researches on the Active Principles of some Aroides.—A. Hébert and F. Heim.—The authors have arrived at the conclusion that the active principles of the *Arum* and allied plants are more or less rich in saponine, according to the season, but the maximum proportion never exceeds 1 part per thousand of the weight of the living plant; that the acrid principle, which has not yet been properly described nor even extracted by any one, is a liquid alkaloid, very similar to conicine; and that it is impossible to detect hydrocyanic acid in the aroids they have worked on.—*Bull. Soc. Chim. de Paris*, No. 13, 1897.

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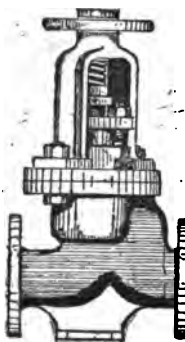
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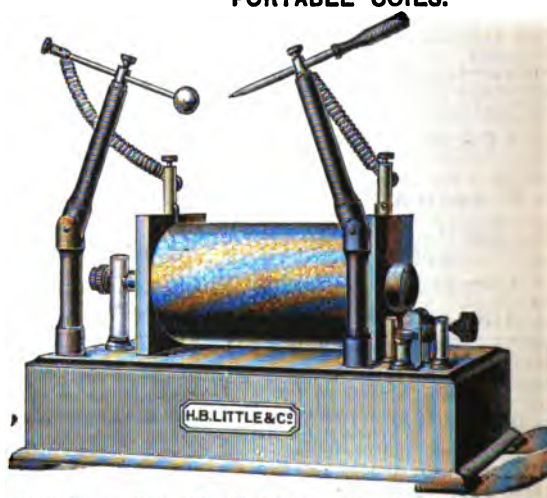
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THE CHEMICAL NEWS.

Vol. LXXVI., No. 1975.

THE PERMEABILITY OF ELEMENTS OF LOW ATOMIC WEIGHT TO THE RÖNTGEN RAYS.*

By JOHN WADDELL, B.A. (Dal. Coll.), B.Sc. (Lond.),
Ph.D. (Heidelberg), D.Sc. (Edin.).

At the meeting of the British Association held last year in Liverpool, a paper by Dr. Gladstone and Mr. Hibbert was read. It was afterwards published in the *CHEMICAL NEWS*, but before I saw their article I had sent for publication, in the same journal, an account of work that I had done along somewhat similar lines, though starting from a different point and undertaken with a different object. My results to a certain extent coincided with theirs, but they were also to a certain extent different, and I wish to bring before the Association some of these differences, as well as some other phenomena connected with the permeability of powders to the Röntgen rays.

My article in the *CHEMICAL NEWS* dealt not only with a number of metals, but with non-metals as well.

In the case of the metals I used mainly oxides and carbonates, whereas Gladstone and Hibbert used formates and acetates. They say that they used the formate because of the low equivalent of the acid radical (CHO_2). As a matter of fact, the carbonate is better from that point of view, as may be seen by considering that 37 parts by weight of lithium carbonate contain as much lithium as 52 parts of the formate, and 100 parts of calcium carbonate contain as much calcium as 130 parts of calcium formate. Of course the disparity between carbonate and acetate is still greater, and equally of course the oxide has a still larger proportion of metal. In any case it is a matter of comparative indifference which of the compounds is taken, since the permeability of carbon, hydrogen, and oxygen is so small.

A point of supreme importance, however, is to define strictly what is meant by saying that one substance is more permeable or has less absorption than another. For instance, Gladstone and Hibbert, in reference to potassium and calcium, say "It was found that, whether tested by the uncombined metals or by their salts, the absorption of the Röntgen rays by the two metals are the same for thicknesses answering to the atomic weights, while they are very different for thicknesses answering to their combining proportions." It all depends on what is meant by "thicknesses answering to the atomic weights," whether the above statement is correct or not. If it means that a plate of potassium 3.9 m.m. thick has the same absorption as a plate of calcium 4.0 m.m. thick, my experiments tend to prove that it is not correct; but if it means that two plates of the same area, and of such a thickness that the weight of potassium would be 0.39 grm. and of calcium 0.40 grm., would absorb equally, I should agree that the statement is within the limits of experimental error. My statement in the *CHEMICAL NEWS* (vol. lxxiv., p. 298) is "The potassium carbonate was of appreciably the same transparency as the calcium carbonate containing the same amount of metal."

I suppose that when the statement was first made that the absorption by metals was proportional to their density, it was intended that equal thicknesses of metal foil should be used. If this law had been true, it would follow that for thicknesses inversely proportional to the density, the absorption would be equal. I believe that this statement

is not very far from correct with regard to the metals of high atomic weight,—say all of those above iron,—but the absorption of all is so large that it is somewhat difficult to establish or to disprove the statement; but there is little difficulty in proving that it does not hold in comparing metals of high atomic weight with those of low atomic weight. Sodium, magnesium, aluminium, are in quite a different class from iron, copper, lead. So much must be admitted by any one who has done work in this connection; but when a comparison is made among the elements of low atomic weight themselves, it seems that there is room for difference of opinion.

I think it must be conceded that the only fair way to test the relative permeability is to have the radiations pass through the same *weight* of the different metals. In order to do this I took pill-boxes of the same size, and put in them such weights of the compounds that they would contain 1.0 grm. or 0.5 grm. of the metal. This was equivalent to taking sheets of metal inversely proportional to their density, and was of course available for many metals which it would be impossible to obtain in sheets in the metallic state. So far as could be determined with metals of high atomic weight, the permeability of equal weights was approximately equal. Tested in this way, however, the permeability of potassium was much less than that of sodium, or magnesium, or aluminium. I found that the difference between lithium and sodium was not nearly so great as between sodium and potassium, and that, if anything, lithium was less permeable than sodium. This is opposed to the statement of Gladstone and Hibbert, who say "that the order of absorption is lithium, sodium, and potassium, while the order of density is lithium, potassium, and sodium." If the weights of lithium and sodium tested, or even the weights of their formates, were in the ratio of 7:23, I should agree with them; but that this is an unfair mode of comparison will be apparent, when it is considered that it corresponds to taking for the test a plate of lithium about two-thirds the thickness of the sodium plate; and no one would think of comparing a plate of aluminium with a *thicker* plate of platinum, which would be a similar experiment.

In view of the variance that appears to exist in the results obtained by Gladstone and Hibbert and myself, I have made a careful re-investigation of the matter, and compared more closely not only lithium and sodium, but beryllium and magnesium, and boron and aluminium. The method of procedure was the same as that employed before, namely, protecting the photographic plate by a sheet of lead in which holes were punched for the pill-boxes. The length of exposure to the Röntgen rays differed in the different experiments, and the battery current and length of spark varied when using the same tube, and two different tubes even were employed. I had found, from previous experience, that it is better to test the permeability of substances in this way than to test their absorptive power by leaving the photographic plate exposed to the full power of the rays, except where covered by the material to be tested.

In the new investigation I took two sets of salts of sodium and lithium, namely, the nitrates and carbonates, and I took varying proportions. In the case of the nitrates I took of sodium nitrate 2 grms. and of lithium nitrate 5.4 grms., $3.2 \text{ grms.} = 5.4 \times \frac{1}{1.7}$, the ratio of the densities, and 2 grms. so as to have equal weights of the two salts: 2 grms. of sodium nitrate and 5.4 grms. of lithium nitrate contain each a little over 0.54 grm. of the metal.

In every photograph the 2 grms. of sodium nitrate was decidedly more permeable than the 5.4 grms. of lithium nitrate, and much more nearly equal to the 3.2 grms. of the lithium salt, which contained only three-fifths as much metal.

I usually considered that the sodium nitrate was more permeable than the 3.2 grms. of lithium nitrate, but the difference was so slight that it was hard to decide. Such

* Read before the British Association (Section B), Toronto Meeting, 1897.

being the case, it naturally follows that the 2 grms. of lithium nitrate was more permeable than the same amount of sodium nitrate.

In the case of the carbonates the weights employed were, of sodium carbonate 1 gm., and of lithium carbonate 2.3 grms., which contained the same weight of metal, namely, 0.43 gm. I had, in addition, two other boxes of lithium carbonate, one containing 1.0 gm., the other 0.7 gm. In this last the weight of lithium is $\frac{1}{7}$ the weight of sodium in the sodium carbonate employed. The difference between the carbonates which contained equal weights of metal was not so marked as in the case of the nitrates—perhaps because of the smaller actual amount of the metal employed; but on all the photographs, except one, which received a short exposure, and which showed several abnormalities, the sodium carbonate was distinctly enough shown to be the more permeable. When the smaller weights of lithium were employed they were more permeable than the sodium carbonate.

Even the 0.7 gm. of lithium carbonate which contains less than 0.14 gm. of lithium had a perceptible absorption, and, if it is considered that this was spread over an area of more than a centimetre diameter, it would appear to be too strong a statement to say that lithium has "next to no absorbent action on the Röntgen rays."

A result that surprised me was that lithium nitrate containing 0.2 gm. of lithium was more absorbent than lithium carbonate containing 0.43 gm. of lithium. The total quantity of lithium nitrate was 2 grms. and of lithium carbonate 2.3 grms.

I therefore tried another experiment in which sodium and lithium nitrate were compared with sodium and lithium carbonate. The salts were taken in such quantities that they contained each 0.27 gm. of metal, that being the amount of sodium in 1 gm. of sodium nitrate. They were placed near the centre of the plate, and placed symmetrically with regard to the Newton's tube; that is, the line joining the cathode and the reflecting anode was in the same plane as the line passing through the middle of the photographic plate, and on each side of this line the nitrates and carbonates were placed. The sodium nitrate was more permeable than the lithium nitrate; there was hardly any appreciable difference between the carbonates, but the sodium was if anything the more permeable. It was no more, however, than might be accounted for by the difference in quantity of the acid radical. The carbonates were very considerably more permeable than the nitrates. I was induced to compare the permeability of substances containing only carbon, hydrogen, oxygen, and nitrogen, with salts of metals. I tried the relative absorption of 1 gm. of dinitrobenzol, 2 grms. of sugar, and 2 of ammonium nitrate. The dinitrobenzol and ammonium nitrate were chosen on account of the considerable quantity of nitrogen in them; in sugar, of course, nitrogen was absent. The sugar was decidedly less permeable than the dinitrobenzol (of which there was only half the amount), and slightly more permeable than the equal quantity of ammonium nitrate. Sodium fluoride and sodium carbonate, containing the same quantity of metal as the ammonium nitrate did of nitrogen, were somewhat less permeable, though the difference was not so great as would, I think, be generally supposed. The sodium fluoride was very slightly less permeable than the sodium carbonate. All of the substances, however, even the 1 gm. of dinitrobenzol, had an appreciable absorbent action.

I also tried potassium and calcium nitrates and carbonates. There was not so much difference between the permeability of the nitrates and carbonates in these cases, partly doubtless because the acid radical is not so large a fraction of the total weight as in the case of lithium and sodium, and mainly that the great absorbent action of potassium and calcium hides that of the carbon, nitrogen, and oxygen.

Since the acid radical has some absorbent action, the greater permeability that I found in the sodium compounds

may be due to their containing a smaller quantity of the acid radical; but one thing is certain, that it is not correct to assert that lithium is of small absorbent action, while indicating that sodium has considerable absorbent power. Though I have not tested it, I should be inclined to expect that lithium chloride containing a gm. of chlorine, which has a very considerable absorbent power, would not show appreciably greater permeability than sodium chloride containing the same amount of chlorine. I am quite ready to admit that, if the permeability of the acid radical of the carbonates is to be tested or compared with that of the nitrates, the lithium salt is better than the sodium salt, because there is less than one-third the quantity of metal in the former as in the latter.

(Note.—Since the rest of the paper was written I have experimented on the absorbent action of the acid radical of the carbonates. I made the comparison with the absorbent action of metallic sodium. For this purpose I took four different quantities of sodium, which I moulded into flat plates to cover the bottom of the pill-boxes. I protected the sodium by a thin coating of vaseline. There were of sodium 0.25 gm., 0.50 gm., 0.75 gm., and 1.0 gm. Of sodium carbonate, I had in another box 1.73 grms., which contained 0.75 gm. of sodium, and of lithium carbonate 1.21 grms., which had the same quantity of the CO_3 radical as the sodium carbonate. The sodium carbonate was less permeable than the 0.75 gm. of sodium, and more permeable than the 1.0 gm., and would I think be about equal to 0.90 gm. of sodium. That would make the permeability of the CO_3 group the same as of 0.15 gm. of sodium, whereas the sodium in the salt is 0.75 gm.—that is, the permeability of the CO_3 group is five times as great as the sodium with which it combines. It was found that the permeability of the lithium carbonate was less than that of 0.25 gm. of sodium, and greater than that of 0.50 gm. of sodium, and would be about equal to 0.40 gm. of sodium. As the CO_3 group has a permeability of 0.15 gm. of sodium, the lithium in the carbonate has a permeability of 0.25 gm. of sodium. In order to compare lithium with an equal weight of sodium, this number must be multiplied by $\frac{1}{2}$, the result being that, if a plate of lithium of the same weight as sodium be compared with it as regards permeability, it is practically equal, being in the ratio of 0.75 to 0.25 $\times \frac{1}{2}$, if the numbers taken above be regarded as correct, as they approximately are. In the same experiment I found that 2 grms. of ammonium nitrate are not so permeable as 0.50 gm. of sodium. This probably accounts for the nitrates being less permeable than the carbonates. The lithium carbonate had a very appreciable absorbent power, the photographic plate being darkened about half as much as when not protected, except by the paper covering and an empty box).

While making the experiments with lithium and sodium I also compared beryllium and magnesium. I used beryllium oxide obtained by heating carbonate which had been obtained from Schuchardt. The magnesium was taken in the form of carbonate. There was 0.53 gm. of the former, and 0.67 gm. of the latter, each containing about 0.19 gm. of the metal.

The difference of permeability between the beryllium compound and the magnesium compound was slight, and in some cases was in the one direction, and in some cases in the other, perhaps depending upon the relative position on the plate or possibly on the length of exposure; but in all instances they were more permeable than the lithium carbonate containing the same amount of metal. It must be remembered that there was a greater quantity of the lithium salt, and though the acid radical has not a very large absorbent power it is not without influence.

Aluminium and boron also were compared, the oxides being the compounds taken: 1 gm. of the aluminium oxide and 1.68 grms. of the boron oxide were employed. The former was obtained by heating ammonium alum, and the latter by heating boric acid.

The aluminium compound was less permeable than

than that of boron, but even it had a greater permeability than the sodium nitrate which contained the same amount of metal (0.54 grm.), and indeed a greater permeability than sodium carbonate containing 0.43 grm. of sodium only.

Boracic acid, which, assuming its formula to be H_2BO_3 , contained the same amount of boron as the aluminium oxide did of aluminium, was, however, not more permeable.

As a result of these experiments, I think it must be admitted that the negative is given to the statement that "In dealing with metals, whether uncombined or in salts, the order of their absorption for these rays is in fact that of their atomic weight, but the amount of absorption increases much more rapidly than the atomic weights themselves."

With the same weight of lithium and sodium the permeability is not far from equal, and for the same thickness of plate of metal sodium is not twice as absorptive as lithium, although its atomic weight is more than three times as great. Nor, in the case of comparing the same thicknesses, does sodium differ as much from lithium as potassium does from sodium.

My present experiments, taken along with the earlier ones, show that a line cannot be drawn between metals and non-metals as regards permeability. Chlorine is less permeable than sodium, but fluorine is more permeable. There seems to be a very rapid increase in absorptive power between the atomic weight of aluminium and that of potassium, and perhaps the same or a slightly less rate of increase on as far as manganese. Above manganese the variation is slight, and below aluminium not very great; but there seems to be the group of elements, carbon, nitrogen, oxygen, with perhaps fluorine and boron, that has a specially low absorptive power. Why carbon should be so much more like fluorine than the latter is like chlorine is a little difficult to say.

A phenomenon which I noticed before, but which I have since more fully investigated, without, however, arriving at what I consider an adequate explanation, is a peculiar granular structure, often exhibited by photographs of powders. In order to see whether the granulation was due to the powder being in lumps, instead of being reduced to an impalpable fineness, I experimented with three grades of powder:—coarse (passing through a ten-mesh sieve but not through a forty-mesh), medium (passing through a forty-mesh but not through a seventy-mesh), and fine (passing through the seventy-mesh sieve). The materials were mainly cryolite and magnesite, and eight different boxes of each were taken as below:—

1. Two grms. coarse powder.
2. Four grms. coarse powder.
3. Three grms. medium powder.
4. Three grms. fine powder.
5. 1½ grms. coarse at bottom, and 1½ grms. fine on top.
6. 1½ grms. fine at bottom, and 1½ grms. coarse on top.
7. 1½ grms. fine and 1½ grms. coarse, mixed.
8. Three grms. coarse, raised 1 c.m. from the photographic plate.

The granulation seemed to correspond with the size of particles, and was quite distinct even through the 4 grms., which made a layer not far from a centimetre thick. When the fine and coarse were mixed, the granulation of the coarse still showed; and when the coarse and fine were separate, the granulation was most distinct when the coarse was at the bottom. When the coarse powder was on top, the appearance in the photograph was very similar to that obtained from the mixed powders. When the box containing the coarse powder was raised a centimetre from the plate, the granulation was very much blurred. This is doubtless due to the overlapping of shadows, and this would account for the greater distinctness mentioned above, when the coarse powder was next the plate and the fine powder on top.

The phenomenon is certainly peculiar, because one would imagine that the particles would lie together in such a way that the average thickness would be the same. This is especially the case when the coarse powder is mixed with the fine, the latter (one would suppose) filling in the interspaces. It is true that a crystal of cryolite about 2.4 m.m. in thickness, placed in 2 grms. of cryolite fine powder, the total thickness being about 7 m.m., was outlined in the photograph, it being slightly less permeable than the powder that would occupy the same space.

Indeed, when the cryolite powder was mixed with about half its weight of calcite powder, the crystal was still fairly well outlined, though not so strongly marked as when cryolite powder alone was used; and I think a mixture could probably be made such that the powder, when pressed into a pretty compact mass, would just equal the crystal in absorbent power. When I had the mixed cryolite and calcite powder it showed a granular structure, though both minerals were so fine that they singly did not show granulation. This, doubtless, was due to an imperfect mixture, little lumps of one or other mineral being unbroken. I fancy that if the mixed powders had been sifted through a fine sieve, the granulation would not have shown.

There was in one box three pieces of cryolite crystal, crossed in such a way that one, two, and three thicknesses were exposed to the rays. The space not taken up by the crystalline pieces was filled in with cryolite powder.

In the photograph the three thicknesses were quite distinctly marked.

I suppose anyone would expect that if ordinary light were passed through a transparent substance, in the same way as was done with the Röntgen rays in the above experiments, no granulation would appear in the photograph. In order to make certain that such is the case, I made use of glass, in fine and coarse powder and in the form of beads of a somewhat larger size. Burning magnesium wire was used for illuminating.

When the photographic plate was developed, it was found, as was expected, that there was no granulation in any case. Of course, ordinary light is refracted and reflected by every particle of the glass, and perhaps we must consider that the different character of the Röntgen rays in this respect is the cause of the granulation shown on the photographic plate; perhaps the particles do not give an average thickness, and the interspaces are distributed irregularly.

A fact which would seem to corroborate this idea was that when a grm. of coarse calcite was mixed with a grm. of medium cryolite and fine magnesite, the dark spaces were larger in the photograph; the spots showing the less permeable calcite being farther apart than when coarse powder only was taken. It does seem strange, however, that the granulation should so nearly correspond to the size of the particles. This was seen most plainly in the case of halite and calcite, perhaps because of the less permeability of these minerals. The bottom layer may give the final impress, and where the particles are least permeable this impress is strongest. I cannot say that I am quite satisfied with this explanation, but no better has yet occurred to me.

The granulation shows so plainly that it could not escape the notice of any one who looks at the photographs in my possession, except where the powder was of impalpable fineness; and if observations made with a photometer did not reveal such a granular structure, it would merely show that the eye is more delicate than the photometer if the eye is assisted, as it was in my experiments, by the sensitive plate being kept covered by the lead sheet. I found this method vastly more sensitive than the one in which the substances formed the absorbent material, the rest of the photographic plate not being shielded from the Röntgen rays.

Kington, Ontario.

THE ATOMIC MASS OF TUNGSTEN.*

By WILLETT LEPLEY HARDIN.

(Concluded from p. 157).

The next line of investigation was to make a number of determinations with material obtained from different minerals and different localities.

Reduction of Tungsten Trioxide obtained from Scheelite from New Zealand.

The oxide was extracted from this mineral and purified in a manner similar to that described under wolframite. No trace of molybdenum was found even before the oxide was heated in hydrochloric acid gas. The results of seven reductions are as follows:—

	Weight of WO ₃ . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	3.41018	2.70410	183.83
2	2.99000	2.37084	183.80
3	3.11613	2.47047	183.67
4	4.32830	3.43118	183.56
5	4.66735	3.70050	183.72
6	4.29620	3.40623	183.71
7	3.39104	2.68885	183.80
8	2.93215	2.32515	183.87

The mean of this series is 183.745. The maximum deviation is 0.20. Considering the number of experiments, this is the most concordant series of results ever obtained by reducing the trioxide of tungsten and weighing the resulting metal. In Experiments 2 and 7, the metal was cooled in a vacuum; in all the other experiments it was cooled in hydrogen.

Oxidation Series.

The metal obtained in the preceding reductions was used in these oxidations. The results of six experiments are as follows:—

	Weight of W. Grms.	Weight of WO ₃ . Grms.	Atomic mass of tungsten.
1	2.70219	3.40775	183.83
2	2.36771	2.98620	183.75
3	2.46705	3.11016	184.13
4	3.42163	4.31472	183.90
5	3.40086	4.28890	183.82
6	2.68249	3.38145	184.20

This series gives a mean of 183.94, with a maximum difference of 0.45.

Reduction of Tungsten Trioxide obtained from Wolframite from Connecticut.

The oxide was obtained from this mineral and purified by the method already described. The details of the work were the same as in similar series which precede. The results were as follows:—

	Weight of WO ₃ . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	3.14520	2.49330	183.58
2	3.10516	2.46141	183.51
3	4.17792	3.31244	183.83

Mean = 183.64

Maximum difference . . = 0.32

Oxidation Series.

The metal was obtained from the preceding reactions.

	Weight of W. Grms.	Weight of WO ₃ . Grms.	Atomic mass of tungsten.
1	2.48088	3.12790	184.05
2	2.44588	3.08318	184.22
3	3.29370	4.15260	184.06

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xix., No. 8.

The mean of these results is almost one-half a unit greater than the mean of the reduction series. It seems that the results from oxidations are invariably higher than those obtained by reduction.

Experiments on Material obtained from Hübnerite from Colorado.

The usual method of purification was used. Two reductions of the trioxide gave the following results:—

	Weight of WO ₃ . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	1.83600	1.45618	184.03
2	4.31878	3.42450	183.81

The metal resulting from these reductions was re-oxidised.

	Weight of W. Grms.	Weight of WO ₃ . Grms.	Atomic mass of tungsten.
1	1.45184	1.83090	183.85
2	3.40470	4.29225	184.14

Experiments on Material obtained from Scheelite from Bohemia.

The oxide was extracted and purified by the usual method. Two reductions were as follows:—

	Weight of WO ₃ . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	2.77363	2.19950	183.89
2	2.13327	1.69120	183.63

The re-oxidation gave:—

	Weight of W. Grms.	Weight of WO ₃ . Grms.	Atomic mass of tungsten.
1	2.18985	2.76060	184.17
2	1.68208	2.12070	184.08

Throughout this work, it had been noticed when tungsten trioxide was heated in a current of hydrochloric acid gas for some time that a considerable sublimate was formed, even when molybdic acid was absent. Enough of this sublimate for an atomic mass determination was obtained as follows:—Tungsten trioxide was heated for some time in a current of hydrochloric acid gas at a temperature of about 400°. The sublimate was removed from the tube, strongly ignited, and gently re-heated in a current of hydrochloric acid gas. The small white sublimate formed did not respond to the test for molybdic acid. The portion left in the porcelain boat was removed from the tube and strongly ignited in the air for a period of ten hours. It was then reduced in a current of hydrogen and the following result obtained:—

	Weight of WO ₃ . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	1.12970	0.89610	184.13

Upon re-oxidation, this metal gave 184.87 for the atomic mass of tungsten.

The results from the sixty-four determinations made in the present investigation show a maximum deviation of one and a half units. A discussion of these results, with a view of arriving at the true atomic mass of tungsten, would be useless. To take the mean of all the results would be entirely unsatisfactory, and yet there seems to be no reason why any one result should be accepted in preference to any other. It will be noticed, in several instances, that three or four consecutive results agree very closely. These different series of concordant results, however, do not agree. The variations in these results are similar in every respect to those in the results of earlier experiments.

Various causes suggest themselves as possible factors in producing these variations. The lower values obtained in the latter part of the investigation are undoubtedly due to a better form of apparatus and a higher temperature.

The reductions were all made in a porcelain boat.

During each determination the boat increased in weight by from 1 to 3 m. grms. It is difficult to determine whether this absorption of tungsten by the boat would affect the results or not. If the tungsten is absorbed as metal it would produce no effect on the results, if not absorbed as metal it would. It was shown in the first part of this investigation that the metal obtained in the reductions contained silica. This may, in part, account for the higher values obtained in the oxidations. In view of these objections to the use of porcelain, a series of reductions were made in which a platinum boat was used. This, however, did not remove the difficulty; platinum absorbs tungsten and tungsten absorbs platinum, and the results obtained were just as variable as those obtained with the porcelain boat.

A series of observations on tungsten trioxide were next made with a view of determining whether or not this compound is suitable for atomic mass determinations.

The first point was to determine how rapidly this compound takes up moisture from the air. Several series of observations were made, and it was found in each case that the absorption of water was inappreciable. The rate at which the water was absorbed is best shown by the following series of weighings of tungsten trioxide, which had been left for several days in the open air. The oxide was first strongly ignited, then placed in a porcelain boat, carefully protected from dust, and left for four days in an open window.

Weight of the oxide at the beginning	5.34600 grms.
" after one day	5.34605 "
" " two days	5.34620 "
" " three days	5.34625 "
" " four days	5.34630 "

From these observations it is evident that no appreciable error can be introduced by the absorption of moisture during the weighing of this compound.

A series of observations was also made to ascertain the action of light on this oxide. A weighed quantity of the material was placed in a desiccator and left for some time in direct sunlight. Weighings made at different intervals showed that no reduction had taken place. In working with this compound it is unnecessary to cover the desiccator with a black cloth.

Upon examining the porcelain tube after a reduction, a slight sublimate was usually noticed. Whether the tungsten trioxide is volatile at that temperature, or whether the moisture formed in the reduction carried mechanically small particles of the oxide from the boat, was not determined. In either case an error is introduced, but in all probability a very small one.

A series of observations was next made to determine whether or not tungsten trioxide contains nitrogen. A number of reductions were made in the usual way and the products set free were conducted through a U-tube containing pure water and a few drops of Nessler's reagent. The oxide used was obtained by strongly igniting ammonium tungstate for two days. Hydrogen was allowed to pass through the reduction apparatus for some time in order to completely remove the air. When the reduction was started, the solution in the U-tube began to assume a yellowish colour, even when the temperature was comparatively low. Before the reduction was half completed, the solution was of a deep yellowish brown colour. In some instances a slight precipitate was formed at the surface of the solution. Several observations were made, and the ammonium test distinctly obtained in each case. A series of blank experiments were made and no colouration was produced. The experiments proved conclusively that the oxide obtained by the ignition of ammonium tungstate contains nitrogen. No attempt was made to determine the quantity of nitrogen present. The oxide obtained by the ignition of metal was also examined and found to contain a trace of nitrogen. Whether the nitrogen in the former oxide was present as an oxynitride or as an ammonium residue, was not determined. If it

exists as an ammonium residue, then hydrogen must also be present. A number of experiments were made by fusing the oxide with lead oxide and also with anhydrous sodium carbonate, with a view of converting any hydrogen present into water. Nothing definite was established, but there were some indications that a small quantity of water was formed. If the nitrogen is present in large enough quantities to affect the atomic mass determinations, it would probably lower the results and also produce variations, for it is not likely that the quantity would be the same in all cases.

In regard to the occlusion of hydrogen by the metal, nothing definite was established. The results obtained by cooling the metal in a vacuum were practically the same as those obtained when the metal was cooled in hydrogen.

It has been shown in the foregoing observations that tungsten attacks the vessels in which the atomic mass determinations have been made, that the oxidation of tungsten is either slightly volatile, or that a small portion is carried mechanically by the water formed in the reductions, and that the supposed trioxide of tungsten contains nitrogen and probably hydrogen. In view of these facts and of the fact that there is no means of determining when the reduction of oxide to metal is complete, and finally, in view of the fact that more than one hundred and fifty determinations have been made of this oxide, and nothing definite established, it is evident that the method usually employed in the determination of the atomic mass of tungsten must be regarded as unsatisfactory.

SEPARATIONS WITH ALKALINE ACETATES.

By HARRY BREARLEY.

(Continued from p. 51).

III.—Cobalt and Manganese from Iron.

In view of the large number of tests it is desirable to make on the separation of each element under various conditions, it is important to choose the most rapid—if at the same time undoubtedly accurate—means available of estimating that element.

With the view of making subsequent tests more intelligible, the method of estimating the separated cobalt is here set forth.

It has been previously noticed that in estimating nickel by means of potassium cyanide and silver iodide, if cobalt be present it will be estimated along with the nickel; but, so far as I can find, nobody has determined whether the titration which serves so accurately for nickel is equally applicable to cobalt. The suggestion, however, is so forcible that it must have been attempted by most people who have made the nickel titration.

In attempting the titration of cobalt solutions, under the conditions previously used, the first noticeable change is the immediate darkening of the solution on adding the cyanide. If the cyanide be further added without unnecessary delay it is seen that the AgI indicator becomes slowly dissolved, and that some time before enough cyanide has been added to combine with all the cobalt. On allowing the just-cleared solution to stand, the AgI turbidity reappears; a few more drops of KCN clear it, further standing causes it to reappear, and so on, but not indefinitely. A point is quickly reached at which the solution can stand for hours without becoming turbid; or if an excess be added in the first instance, there is no turbidity on standing. In this latter way—that is, by adding cyanide in excess, allowing to stand awhile, and going back with silver nitrate—it is easy to obtain consistent values when titrating similar amounts of cobalt.

The darkening of the solution just noticed is not permanent. The usual change is from the deep golden brown to a salmon-coloured liquid which very gradually

bleaches. The colour changes, however, are not always the same, except in identical liquids treated alike. The precise colour effect of each reagent has not been determined, but the addition of potassium iodide before or after the cyanide gives distinctly different tints.

It is practicable to replace ammonium by the corresponding sodium salts, and to make the final alkalinity with sodium carbonate. This change gives us a colouration which is neither so deep nor so variable, and the disappearance of the precipitated carbonate on adding cyanide is some indication of the progress of the reaction. Personally, this soda modification has proved eminently satisfactory, almost rivalling in delicacy the cyanide titration of nickel; in other hands it has not behaved so well. A comparison of soda and ammonia salts, kindly made by a friend, led him to prefer the latter.

Unfortunately whether soda or ammonia is used, the dilution of the solution titrated is not without influence. Results from two sets of solutions are appended.

TABLE VIII.

	Volume of solution. C.c.				
	120.	200.	300.	400.	
Ammonia..	17'84	17'52	16'92	—	c.c. KCN
Soda ..	18'22	17'93	17'58	17'14	" "

The tests in each set varied only in volume. They contained the same total quantity of free alkali, so that the proportion of this reagent is less in the larger volumes. This difference would tend to give more concordant values.

Lacking at present the recommendation of extended use in various hands, it was deemed undesirable to use this method alone and unchecked. Corresponding titrations were therefore made here and there according to Winkler's method (see "Fresenius's Quant. Anal.," or "Sutton's Vol. Anal."), which is regarded as "thoroughly satisfactory for technical purposes" at least. It may be worth while to notice that, contrary to expectations, there was no noticeable trouble experienced on account of the organic (acetic) acid.

Any error due to traces of manganese in the bar iron was exactly balanced by treating a sample—lacking cobalt—exactly like the test, and standardising the permanganate or cyanide in an equal portion of the filtrate.

The details of the separation to be now applied to cobalt are the same as those followed with nickel and iron, and presuming an acquaintance with the Ni titration, that for cobalt may be summed briefly:—The idea is to get an excess of cyanide into the solution, but not a large excess. Make the solution alkaline, and then, without unnecessary delay, add cyanide to completion or thereabouts; add the indicator ($KI + AgNO_3 + Am_2SO_4$ or Na_2SO_4), and then, if necessary, enough cyanide to clear the solution and 2 or 3 c.c. in excess. I prefer to use a very dilute solution of silver nitrate—so dilute that 10 c.c. is only equal to 1 c.c. KCN where about 16 KCN equals 0.01 gm. cobalt. The KCN is standardised with a similar amount of cobalt in exactly the same way, and the two are allowed to stand awhile—say, fifteen minutes. If they are then perfectly clear,* silver nitrate is added until the turbidity reappears. This is most satisfactorily accomplished by allowing the nitrate to run down the side of the flask. A very copious turbidity is soon formed on the surface of the solution, and the readiness with which this disappears on shaking is some indication of the amount of silver nitrate yet to be added. It is easier, too, in this way to note the first appearance of the turbidity. 2 c.c. of the silver nitrate makes a decided difference in half a litre of solution; 2 c.c. $AgNO_3$ equals 0.2 c.c. KCN, equals 0.00013 gm. cobalt.

Samples containing 1 gm. (separations will always be

* A slight turbidity, due to traces of manganese, should be filtered off if it is so decided as to prevent the AgI turbidity being readily recognised.

from 1 gm. unless otherwise stated) of iron and 0.1 gm. of cobalt gave the values shown in—

TABLE IX.

	Method of titration.	
	KCN.	Winkler.
Ammonia salts	0.0995	—
Soda salts	0.0997	0.1015

Nor is the separation less perfect with other proportions of cobalt. In parallel columns there are arranged some results by Messrs. Leffler and Jervis, to whom my best thanks are due. Apart from their confirmatory value, such results are testimony to the fact that the method may be satisfactorily worked without lengthy experience. Their results are with ammonia salts throughout, and on mixtures of bar iron and cobalt nitrate. My own results are with soda salts throughout.

TABLE X.

Added. Grm.	Cobalt found.		
	Brearely.	Leffler.	Jervis.
0.0200	—	0.0196	0.0198
0.0300	—	0.0294	0.0301
0.0500	—	0.0496	0.0508
0.1000	0.0997	0.1001	0.0995
0.2000	0.2006	—	—
0.3000	0.2982*	—	—

* Titration irregular.

For the rest, it remains only to notice a few variations similar to those observed with the preceding metal. To prevent repetition, the corresponding tables in the nickel series will be placed in brackets so that the details of the tests can be referred to.

Further addition of acetate may be made to the heated solution (p. 50, col. 1).

TABLE XI.

	Co added.	Found.
Soda salts	0.1000	0.1003
Ammonia salts	0.1000	0.0997

Prolonged boiling introduces no error (VI.).

TABLE XII.

Boiled.	Cobalt added.	Cobalt found.
15 minutes	0.1000	0.0997
30 "	0.1000	0.1000

Deservedly or otherwise, the separation of cobalt from iron by means of acetates is less favourably looked upon than the corresponding separation of nickel. Thus, T. Moore (CHEMICAL NEWS, lxxv., 75), who seems to have made a special study of these two elements, says:—"A very short experience will suffice to demonstrate that the basic acetates of iron must be re-precipitated at least four times before one can be assured that the separation, so far as cobalt is concerned, is complete."

Whether cobalt is less readily separated from iron than is nickel would be seen by adding varying amounts of acetate in excess and, making every other condition alike, noticing in which the percentage recovery fell the more rapidly. The results of such experiments are arranged in Table XIII. The agreement between the temperatures of turbidity should not be overlooked. It afforded additional evidence that the two sets of solutions were very similar. The minimum amount of acetate for solutions made up as these were would be about 12 c.c.

TABLE XIII.

Acetate. C.c.	Percentage recovery of—		Respective Temp. turbidities.
	Nickel.	Cobalt.	
20	99.0	99.04	72°, 70° C.
50	95.2	97.5	60°, 59° C.
100	90.0	93.8	53°, 54° C.

This table can claim to answer the question at issue only for the particular *modus operandi* used, which was that generally adopted for the previous separations. It does not necessarily follow that the same order would hold whatever variations might be introduced, though for two so closely related elements as nickel and cobalt the result may perhaps be considered generally true.

A table similar to XIII. will be given later embracing all the metals whose separations from iron have been attempted.

Separation of Manganese.

A previous paper on the estimation of manganese in *spiegels* (CHEMICAL NEWS, lxxv., 13) makes it needless to repeat any array of experiments. This circumstance affords us an opportunity of looking about more casually than it might otherwise be thought proper to do. The paper mentioned contains an error of some moment; a few words will set things right. The use of ammonium acetate is restricted, because in heated solutions salts of ammonia react with the permanganate in the subsequent titration. It may be noticed that as much as 10 c.c. of strong ammonium acetate is used to precipitate the one gm. of iron; this is several times larger in amount than the soda acetate used, or than it need be. This correction made, the danger of the permanganate reaction would be lessened.

It is noteworthy that while the separation of nickel and cobalt from iron by means of acetate are commonly deprecated, and are fast disappearing from the arena of usefulness, the separation of manganese by these means has, on the whole, and particularly for low percentages, been well spoken of. This argues in a general way that the former metals are less accurately separated from iron under similar conditions than is manganese. Indeed, if a series were arranged parallel with those first given (Table XIII.), it would be seen that as perfect a separation accompanied the larger amount of acetate as the smaller. In fact, Mr. Jervis has, in this laboratory, increased the acetate to 200 c.c., and finds still a perfect separation. The instructions, therefore, for separating iron and manganese perfectly and without re-precipitation, may be condensed into a word—ditto.

One frequently hears the use of soda salts in the gravimetric estimation of manganese justified by the belief that they give a better separation than the corresponding ammonium salts. There is no doubt but that either will give a perfect separation when used sparingly enough. That there could be any question of their comparative merits reminds one of the careless way in which the acetates are generally used—as though an excess were of no moment, or perhaps improved the separation.*

The relative efficiency of these two acetates was tested by using such large excesses as to cause imperfect separations. As the point is not an important one, it may be sufficient to notice that the result was so slightly in favour of ammonium acetate that they may be regarded as practically equal. It is to be feared that the higher results with soda acetate are sometimes due to the well-known difficulty of washing away the non-volatile precipitant.

It appears plainly that alkaline acetates have been ill-spoken of and neglected, not because of any inherent shortcoming in the reagents themselves, but on account of the thoughtless way in which they have been used. There are other highly-prized reagents which would fail under like treatment. That I may not be accused of unduly magnifying this error of excessive acetate, I quote below a few instances from such recently described methods as retain this means of separation. The figures in brackets give the approximate volume if diluted to such strength as is used throughout this investigation.

* Parry and Morgan (*Industries*, 1893; also CHEMICAL NEWS, lxxv., 308), respecting the separation of nickel and cobalt from iron, recommend that the solution be "made neutral with soda carbonate and the iron precipitated by the addition of a large excess (italics copied) of soda acetate and the solution boiled for some time" (italics not copied).

Parry and Morgan (CHEM. NEWS, lxxv., 295, 1893)—250 c.c. hot ammonium acetate, strength not stated.

Arnold ("Steel Works Analysis," 1895)—Half a gm. of *spiegel* is precipitated with 20 c.c. [260] ammonium acetate. And 25 c.c. [more than 250 c.c.] saturated solution of sodium acetate is used to separate 0.1 gm. nickel from similar quantity of iron.

Dittmar ("Quant. Chem. Anal.," 1887)—"Add acetate of ammonia solution (not too little)."

Phillips ("Engineering Chemistry," 1891)—later edition not available—Half gm. of *spiegel* precipitated with 20 c.c. [290] 5 E ammonium acetate.

Hiorns ("Practical Assaying," 1892)—1.3 grms. steel 20 c.c. [260] acetate.

These large excesses become more curious when it is noticed that (with the exception of Phillips) strong emphasis is usually laid on the necessity of neutralising with great exactness.

The last writer (Hiorns) says:—"If the basic acetate precipitate be dark red an insufficient amount of acetate has been added, and more of that salt must be introduced" (p. 397). I have previously heard this remark made to beginners in laboratory practice, though it is rarely repeated in text-books. Altogether apart from the peculiar appearance due to iron in the ferrous state, the basic acetate may vary from a fine compact brick-red precipitate, which remains long suspended in the solution and can be eliminated only by repeated filtration through ordinary paper, to an open flocky purple-brown precipitate, which settles readily and leaves a crystal clear solution. The former occurs in presence of large volumes of acetic acid, the latter by precipitating with little more than minimum acetate. Similar precipitates, and any variety between, can be produced with other amounts of acetate. The inference is that the colour of the precipitate alone is of no value in determining whether enough acetate has been added or not.

(To be continued).

NOTES ON THE ESTIMATION OF SILVER IN SILVER-PLATING SOLUTIONS.

By T. J. BAKER.

I. THE method of weighing the precipitate obtained with HCl is untrustworthy in the case of old solutions, unless precautions are taken to separate the copper cyanide and other impurities precipitated along with the silver chloride.

II. The method of fusing the impure silver chloride with sodium carbonate and nitre is often attended with the disadvantage of small globules of silver adhering to the crucible.

III. The following method devised by the writer avoids the disadvantages referred to above:—

Precipitate say 50 c.c. of the solution by boiling with slight excess of nitric acid. The precipitate will contain all the silver as silver cyanide, together with copper cyanide and other impurities. Wash and dry the precipitate.

Burn the filter-paper, and wrap up the ash, together with the precipitate, in assay lead. Compress into a small bulk, and cupel, employing the usual precautions of duplicate assays and checks.

Note.—Silver cyanide is completely reducible to metallic silver by simple ignition, and the effectiveness of cupelling the precipitate depends upon this reaction.

The method has been attested in the metallurgical laboratories of the School, and has given uniformly good results.

Electro-Metallurgical Laboratory,
Birmingham Municipal Technical School,
September 2, 1897.

ON THE
PERMEATION OF HOT PLATINUM BY
GASES.

By WYATT W. RANDALL.

IN connection with a research which has been in progress for some time, and in which the attempt is being made to prepare absolutely pure hydrogen for spectroscopic examination, the author has endeavoured to free the gas from impurities by filtering it through a diaphragm of hot platinum. The spectroscopic results obtained will be published later; it seemed worth while, however, to give at this time a short account of the work which has been done in the past in the matter of the permeation of metals by hydrogen, and also of the measure of success which has attended the attempt to free the gas from impurities by the method mentioned.

The fact of the permeability of hot platinum by hydrogen was first announced by Deville and Troost (*Compt. Rend.*, lvi., 977, 1863; *Phil. Mag.* [4], xxvi., 336; *Abstr. Fsb.*, 1863, 23; *CHEM. NEWS*, vii., 294). When hydrogen was introduced into a hot platinum tube surrounded by one of porcelain filled with air, the former gas passed through the walls of the metal tube, but no passage of air into the inner tube could be detected. Later papers by the same authors (*a*, *Compt. Rend.*, lvii., 894, 1863; *b*, *Ibid.*, lvii., 965; also *Ann. Chem.*, Liebig, cxxx., 254; *c*, *Compt. Rend.*, lix., 102, 1864; *Abstr. Fsb.*, 1864, 89; *Phil. Mag.* [4], xxviii., 229; *CHEM. NEWS*, x., 57) established the results claimed, and showed that iron also was permeable by certain gases at high temperatures.

A year or two later Graham (*Phil. Trans.*, clvi., 399, 1866; *Phil. Mag.* [4], xxxii., 503; *J. Chem. Soc.*, xx., 257; *Abstr. Proc. Roy. Soc.*, xv., 223) repeated the experiments of Deville and Troost, taking, however, greater precautions to avoid error. His method differed in that, by means of a Sprengel pump, a vacuum was maintained within a platinum tube, which was closed at one end, and which was heated in an atmosphere of hydrogen. The gas was found to penetrate rapidly into the interior of the tube. When air was substituted for hydrogen, practically no gas was delivered by the pump, but Graham was of the opinion that air did penetrate to a slight extent. As, however, the connections of his apparatus were not above suspicion, and as he employed air which had been dried by sulphuric acid alone, the evidence for the penetration by air is by no means convincing. At the temperature employed, any water-vapour present in the air would probably have been at least partially dissociated and free hydrogen have been formed. The same objection might hold good for all his experiments in this series. When carbon dioxide was tried 0.3 c.c. of gas was collected by the pump, and "an indeterminate small portion of this was condensed by baryta water and appeared to be carbonic acid." In view of the power of carbon dioxide to cling to the walls of a vacuous glass tube, and to be but slowly removed, the evidence here, again, is inconclusive. Negative results were obtained with chlorine, hydrochloric acid gas, steam, and ammonia. When coal-gas containing hydrogen, methane, carbon monoxide, and ethylene was employed, hydrogen penetrated, but no evidence of carbon compounds could be obtained on exploding the gas collected with oxygen. Graham concluded that a little nitrogen might penetrate the platinum wall, provided hydrogen was passing through at the same time in the opposite direction, since he obtained evidence of such penetration when the tube was filled with hydrogen and heated in an atmosphere of nitrogen.

In accordance with conclusions arrived at by a study of the passage of gases through various septa, Graham assumed that the penetration of platinum by hydrogen was due to the liquefaction of the gas on the surface, the diffusion of the condensed gas through the material of the wall, and the evaporation of the liquid from the inner surface of the tube into the vacuum.

Nearly all research in this field since the publication of these results has had to do with the nature of the occluded gases, and not with questions of penetration considered by itself. Morse and Burton, however, have shown* that the hydrogen contained in the flame of an ordinary blast-lamp will penetrate a platinum vessel heated by that means; and Ramsay, in studying the phenomena connected with the passage of hydrogen through a palladium septum, has found (*Phil. Mag.* [5], xxxviii., 206, 1894) that this power was apparently possessed by no other gas examined. As palladium is permeable to a far higher degree than platinum, it would naturally be assumed that the latter metal would resist the passage of all gases except hydrogen completely.

In his studies of the properties possessed by hydrogen when occluded in platinum and palladium, Graham found that the metals could be more highly charged with the gas by making them serve as the negative pole of galvanic cells, than by heating them and allowing them to cool in an atmosphere of hydrogen (*Proc. Roy. Soc.*, xvi., 422, 1868). "The occluded hydrogen," he says, "is certainly no longer a gas, whatever may be thought of its physical condition." Oxygen was not occluded when the current was reversed. The evidence, he considered, was on the whole against the view that chemical combination takes place between the metal and the gas. Careful examination of the change in volume suffered by palladium and its alloys on absorbing hydrogen led to the view that the gas was converted into a metal-like substance and formed a species of alloy with the palladium (*Proc. Roy. Soc.*, xvii., 212, 500, 1869; *Compt. Rend.*, lxviii., 1511). Hydrogen in this particular condition—"Hydrogenium," as Graham calls it—is magnetic, shows electric conductivity, and possesses some tenacity. Its density varies: in palladium alloys it is 0.711–0.7545; in palladium itself, 0.854–0.872.

This conclusion was, however, challenged in the more recent work of Berthelot (*Compt. Rend.*, xciv., 1377, 1882; *Ann. Chim. Phys.* [5], xxx., 519), who, as a result of thermo-chemical investigations, returned to the view that definite complex hydrides of palladium and platinum are formed. The results obtained by Thoma (*Ztschr. Phys. Chem.*, iii., 69, 1889) had to do with the absorption of the gas considered as a case of solution capable of supersaturation. The conclusions of Berthelot have been offset by a research by Mond, Ramsay, and Shields (*Phil. Trans.*, 186 A, 657, 1895; *Abstr. Proc. Roy. Soc.*, lviii., 242) upon platinum-black, in which doubt was cast upon the purity of the material used by the French chemist, and evidence given to show the untrustworthiness of his thermo-chemical data for establishing the condition of the occluded gas.

The method of spectroscopic examination seemed capable of yielding so much more accurate results in determining the presence of foreign gases in the filtered hydrogen than any of the analytical methods to my knowledge used hitherto in this work, that it seemed worth while to put it to trial. Tubes filled with gas were examined in the Physical Laboratory of this University, with the aid of a large concave grating specially prepared for such work, and photographs were made of each in turn. For their kindness in making these examinations I am indebted to Dr. J. S. Ames and Mr. W. J. Humphreys.

The essential features of the apparatus used in the production of the gas to be tested are represented in the accompanying figure. It consists of a platinum tube, A A', about 350 m.m. long, and of about 3 m.m. internal diameter, closed at one end and sealed at the other, by means of fusible glass, into a soft-glass tube, which is in turn connected with the sparking-tube D and the Töpler pump. The closed end of the platinum tube projects into a piece of hard-glass tubing, B B', through which a current

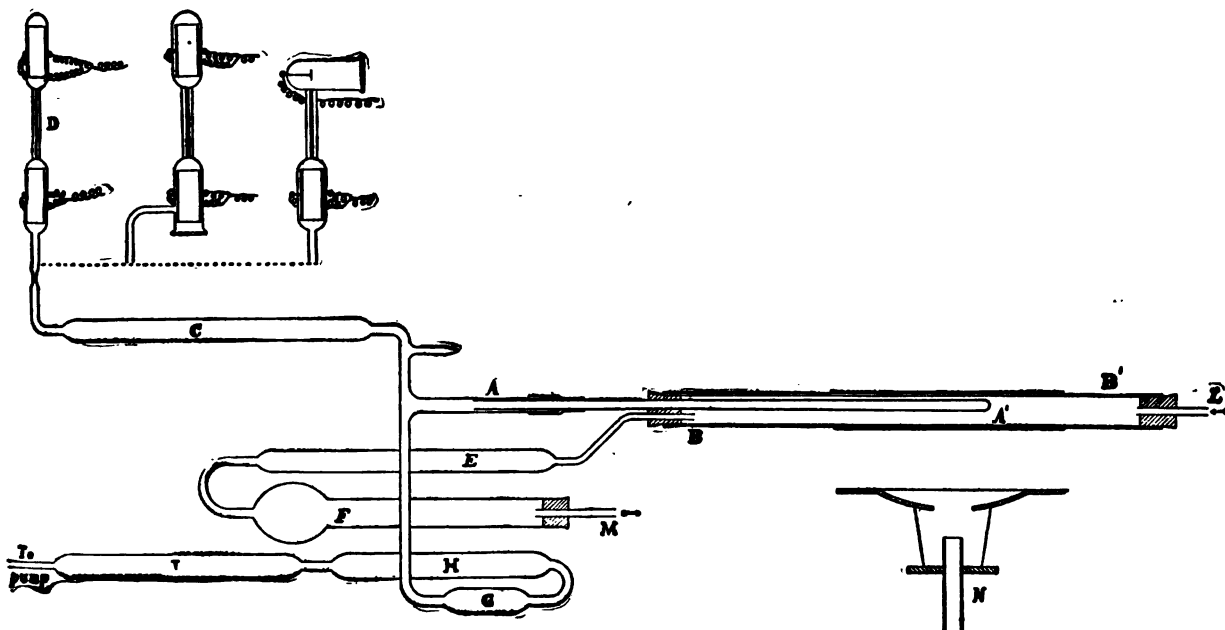
* *American Chemical Journal*, x., 148 (1888). It is to Professor Morse that I owe the suggestion of employing a platinum septum in this work.—W. W. R.

of dry gas, entering at L, could be passed, while at the same time the platinum tube can be heated by means of a Bunsen burner, N, below the combustion-tube. The latter is covered for a portion of its length with a layer of thick copper foil to distribute the heat, and, with the aid of asbestos-board screens, the temperature of the two tubes can be raised to a white heat. A plug of asbestos was pushed into the annular space between the two tubes to give support. A short piece of black rubber is drawn over the place where the platinum tube is sealed into the glass tube, is wired on and well shellaced, to guard against possible leakage from fracture.

The tube c is filled with phosphorus pentoxide. The gas entering at L has been made to pass through sulphuric acid, calcium chloride, soda-lime, and, finally, through a long tube filled with phosphorus pentoxide. It passes out at s through the guard-tubes x containing the pentoxide, and r containing calcium chloride and soda-lime. When hydrogen was the gas employed, it was first passed through four wash-bottles filled with acid, and two filled with alkaline permanganate solution, and then through a tube filled with red-hot copper turnings, on its way to the drying-tubes. It was hoped the latter device would free the gas from any admixture of oxygen, whether present as a constituent of air, or given off from the permanganate solution (see footnote at end of paper).

of course taken to have the air as dry as possible, in order to avoid the presence of hydrogen through the dissociation of water-vapour. The vacuum within the metal tube could be maintained apparently indefinitely under these conditions.

The tubes were filled with hydrogen as follows:—The carefully cleansed tube was sealed to the apparatus, and, when the air had been completely exhausted, was left for twenty-four hours or more; by this time all moisture was probably removed by the phosphorus pentoxide. By continuous sparking and exhaustion the gas occluded by the electrodes was then removed, and a condition of brilliant green fluorescence readily obtained. On raising the mercury in the Töpler pump so as to compress all the gas in its bulb and tubes into a space of not more than a fraction of a cubic centimetre, and at the same time noting the change, if any, which took place in the height of the mercury column in the delivery-tube,—in other words, on using the pump itself as a McLeod gauge,—it could be shown that the pressure within the exhausted space was not more than one or two millionths of an atmosphere. Connection between the pump and the rest of the apparatus was then cut off by turning a glass tap, and hydrogen was admitted slowly into the sparking-tube by heating the metal tube while a current of dry purified gas was circulating about it. When the pressure inside the



The sparking-tubes were of several forms, at first with wire electrodes, afterwards with cylindrical electrodes of aluminium tubing or foil. In some cases photographs were made of the spectrum of the light emitted through the walls of the capillary tube; in others, of that observed "end on" through a disk of quartz sealed as a window to one of the bulbs. In one case the tube was specially constructed so that the light observed through the window was that produced at the electrode and not that in the capillary tube; the light thus obtained was, however, so weak that no photograph could be made of the spectrum.

Having demonstrated the tightness of the apparatus, and the fact that at the temperature employed hydrogen is capable of permeating the walls of the platinum tube, experiments were made which afforded evidence that a similar power of penetration is not possessed by oxygen or nitrogen. Air was drawn through the apparatus while the metal tube was maintained at a white heat. Care was

sparking-tube had reached about 1 c.m., the heating was discontinued and the apparatus exhausted as before. The process of filling and exhausting was repeated from three to six times, after which the tube was filled to the desired pressure—which was between 3 and 8 m.m.—and was sealed off. In some cases, as an extra precaution, the tube was enclosed in a copper air-bath, and heated during the process of exhaustion to a temperature of 150° to 200°, the sparking being meantime continued.

It may be as well to say that the passage of the hydrogen into the exhausted space was very slow as compared with what the statements of Graham had led me to expect. It may be that the tube employed by me was thicker and more thoroughly hammered, and that the temperature obtained was not so high. At all events the pressure in the sparking-tube rose only at the rate of about a millimetre in three to five minutes, the volume of all the tubes and connections open to the gas being about 100 c.c. On this account the time required to cleanse,

exhaust, and fill the tube in the manner described, waiting in each case for diffusion to equalise the pressure throughout the long system of tubes, rendered the work very slow and tedious, especially as only a short time was available on any one day to be devoted to the work.

The sparking-tubes had at first, except for the tube c, been in direct connection with the Töpler pump and mercury gauges. On photographing the spectrum it was found that the gas was contaminated with the vapour of mercury. To avoid this difficulty the tubes g, h, and i were introduced between the pump and gauges and the system to be exhausted. i is a long tube filled with powdered sulphur, h a similar one containing bright copper turnings, and g contains gold-leaf. h and i are shown in the figure much shorter in proportion than they are in reality. So perfectly do these guard-tubes serve their purpose, that after one year the gold-leaf shows no signs of amalgamation. The copper has apparently held back all sulphur-vapours from the sparking-tubes, whereas the mercury in the manometers have become fouled from not being protected in its turn by copper. No trace of mercury-vapour has been detected in the gas contained in any of the sparking-tubes since the precautions mentioned were taken to exclude it.

Results.

A careful examination of the photographic negatives obtained, when the hydrogen produced as described and contained in the sparking-tubes was used as the source of light, has failed to reveal the presence of any other substance—unless the so-called "compound" spectrum is to be considered an indication of such contamination. The purified hydrogen drawn through the combustion-tube b b' probably frequently contained nitrogen, but no signs of that gas could be detected in the spectrum of what had passed into the inner tube. If the compound spectrum is due to the presence of water-vapour, it is not clear how it can be eliminated, no more efficacious drying agent than phosphorus pentoxide being available.

It may be well to mention here that the results obtained in the examination of the spectrum of hydrogen purified as described in this paper, do not seem to agree with those obtained by Messrs. Trowbridge and Richards (*Am. Journ. Sci.* [4], iii., 118; *Phil. Mag.* [5], xliii., 137, 1897). According to these authors, an oscillatory discharge from their powerful storage-battery through hydrogen did not yield the compound spectrum, whereas the direct discharge produced the compound spectrum as usual. In the experiments performed with hydrogen, as described in this paper, the discharge was that of a large Ruhmkorff coil, sometimes with and sometimes without a Leyden jar in the secondary circuit and with considerable variations of current in the primary coil. In no case was the compound spectrum absent. Messrs. Trowbridge and Richards do not mention the character of the tubes they employed, nor give any account of the method of purification used for the gas, or that of cleansing and filling the tubes. It is just possible that were these data available, some explanation of the difference in our results might be forthcoming. It is true that the pressures used in this work were on the average a little higher than those employed by Trowbridge and Richards, but not to an extent, in all probability, sufficient to account for so marked a discrepancy.

As has been stated, oxygen and nitrogen do not penetrate hot platinum under the conditions described above; it remained an interesting question to determine whether this was due in any degree to the relatively high specific gravity of these gases as compared with hydrogen. Accordingly an experiment was made to see if marsh-gas, with a density only half that of oxygen, would penetrate into the inner tube. A large gasometer was filled with the gas prepared by heating a mixture of dehydrated sodium acetate and soda-lime, and washed with water and acid sodium sulphite solution to remove the vapours of acetone. After standing several weeks in contact with

water in the gasometer, it was passed again through a solution of the sulphite, through soda-lime, calcium chloride, and sulphuric acid, to mix with the purified hydrogen as it entered the tube filled with hot copper turnings mentioned above.* After the air had been removed, and a gas mixture, consisting of about four-fifths marsh-gas and one-fifth hydrogen, was being passed through the combustion-tube b b', the latter was heated. Gas passed very slowly into the sparking-tube; after about three hours the pressure had risen to only about 3 m.m. Examination of the gas by means of a large prism spectroscopie failed to show the presence of any carbon compound; but, on account of the low pressure the light was too feeble to give any result with the photographic method, even after a very long exposure, and so this experiment will still require further and more accurate confirmation.

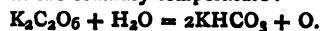
It is proposed to continue this work with hydrogen and other gases.

Since this paper was written a note has been published by Ramsay and Travers (*CHEMICAL NEWS*, lxxv., 253), in which they describe their attempts to pass helium and argon through septa of platinum, palladium, and iron, under conditions similar to those described in this paper. No evidence of such penetration was, however, obtained. —*American Chemical Journal*, xix., No. 8.

ON A NEW CLASS OF OXIDISING SUBSTANCES: THE PERCARBONATES.

In a recent number of the *Zeitschrift für Electrochemie* MM. Constam and A. von Haussen announced the discovery of a new series of compounds which are of very great interest. We know that on electrolysis the alkaline carbonates, M_2CO_3 , we obtain hydrogen and the hydrate of the constituent base at the cathode, and at the anode oxygen and carbonic acid, which re-combines with a part of the base to form bicarbonate.

The authors have observed that if we electrolyse a saturated solution of carbonate of potash, and gradually lower the temperature, the disengagement of oxygen gradually diminishes at the anode, and finally ceases completely at about $-10^\circ C$. And further, instead of a crystalline bicarbonate being formed, we have a bluish amorphous powder, shown by analysis to consist of $K_2C_2O_6$; this is percarbonate of potassium. We can thus explain its formation: the carbonate of potassium in saturated solution first of all becomes dissociated into ions K and KCO_3 ; when electrolysis intervenes, the two ions KCO_3 unite, to form the body $K_2C_2O_6$. The phenomenon does not occur in dilute solutions, as the carbonate of potassium splits up into the ions K and CO_3 . The percarbonate obtained in the above manner should be quickly thrown on a filter, and dried over phosphoric anhydride. It is very hygrometric, and decomposes water at the ordinary temperature:—



When gently heated it decomposes according to the following equation:—



In the presence of oxidisable matters it acts as an oxidising agent. But it can also act as a reducing agent:—



* It is not generally known that comparatively large quantities of oxygen are set free when hydrogen is made to bubble through potassium permanganate solution. Unless care is taken to remove this oxygen, say with red-hot copper and a drying agent, an opportunity is given for a very serious error in all experiments where pure hydrogen is desired. This reduction of permanganate by hydrogen is now under investigation in this laboratory by Prof. Morse.—W. W. R.

From these reactions the authors conclude that this new body is in reality the neutral carbonate of a higher oxide, peroxide of potassium. Besides it produces, like the higher alkaline oxides and the alkaline earths, peroxide of hydrogen in the presence of acids. — *Revue Générale des Sciences*, No. 17, 1897.

CORRESPONDENCE.

SUPPOSED NEW ELEMENT WITH IRON.

To the Editor of the Chemical News.

SIR,—With reference to the article by Mr. G. G. Boucher (CHEMICAL NEWS, vol. lxxvi., p. 99), on the discovery of a possible new element with iron, I should like to point out that the reactions given by him in that article for his unknown metal are almost identical with those of molybdenum, the only exception being with Na_2SO_3 , which with dilute solutions gives no perceptible change as in his case, but with a fairly strong solution gives a blue colouration on boiling, while with SnCl_2 , in the case of the metal separated from iron, no change takes place; with the metal produced from boiler dust, SnCl_2 gives the usual molybdenum colourations of dark blue in the cold and brown on boiling with HCl ; and further, the most characteristic reaction for his unknown metal, viz., the intense blue colour produced by evaporation with sulphuric acid, is also one of the more characteristic tests for molybdenum, and therefore—seeing that from Mr. Boucher's description of the process for separating his unknown metal no steps are taken to remove molybdenum should such be present—I would suggest that this metal may prove to be molybdenum.—I am, &c.,

CHARLES H. JONES.

Laboratory, Minas de Rio Tinto, S. Spain,
September 3, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxv., No. 10, September 6, 1897.

The Minister of Public Instruction and the Fine Arts addressed to the Academy the amplification of the decrees by which the President of the Republic authorises the Academy to receive the donations offered by Henry Wilde for the foundation of an annual perpetual prize of 4000 frs. to be awarded to the person whose discoveries or researches in astronomy, physics, chemistry, mineralogy, geology, or experimental mechanics shall be judged most worthy of reward.

The Magnetic Deflection of the Cathodic Rays and of the X Rays.—G. de Metz.

Action of the X Rays on the Luminescence of Gases.—A. de Hautpierre.—A tube containing a gas at a low pressure becomes luminous under the action of electric vibrations, and becomes luminous at a much higher pressure if submitted to the action of the X rays.

Revue Générale des Sciences Pures et Appliquées.
No. 15, August 15, 1897.

This number contains no original matter of chemical interest.

Bulletin de la Société d'Encouragement pour l'Industrie Nationale. Series 5, Vol. ii., No. 7, July, 1897.

This number contains an account of the annual general meeting of the Society, which took place on June 25th under the presidency of M. Mascart, who delivered an address. The reports of various committees were presented and a number of medals conferred.

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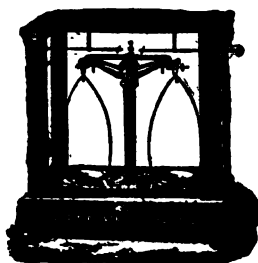
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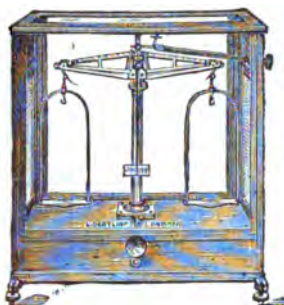
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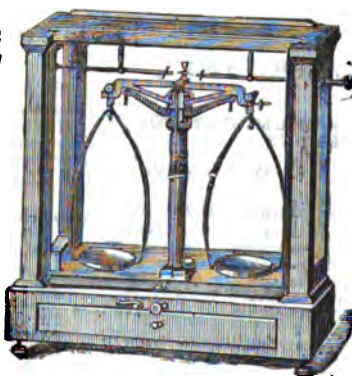


FIG. 3a.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1976.

OCT 18 1897

THE GOVERNMENT LABORATORY.

By CHARLES A. MANSFIELD.

THE new Government Laboratories, which have been upwards of two years in construction, were formally opened on October 1st. They are situated in close proximity to King's College Hospital and to the rear of the New Bankruptcy Court, and occupy a site of 7900 square feet.

Outwardly the building is a plain one, of red brick; the rooms (which occupy three floors and a basement) are lofty and well lighted, their walls of faced bricks are white with a dado of pale blue; the floorings are of cement overlaid with pine blocks. Ventilation is effected by a fan in the basement, the means of artificially heating and lighting the establishment being steam and electricity respectively. There are three distinct water systems,—one in connection with the Water Company's main, a second supplied by a reservoir situated under the basement, and a third of cooled water, a small reserve of which is kept in a tank near the roof. The pipes conveying these services, as well as those of gas and steam, are easily accessible and naked throughout their length, and painted in different colours according to the use to which they are put. Those of each bench are separately connected with their several mains, so that, should any leakage occur in them, they can be thrown out of use without other than local inconvenience. Dirty waste liquid is conveyed away by V-shaped wooden troughs covered with lead thickly coated with paint; the difficulty of corrosion accompanying the use of leaden pipes for this purpose is thus avoided.

The basement is mainly devoted to store- and engine-rooms. A sample receiving room contains an ingenious bottle-washing apparatus. The steam leads of the boiler are fitted with reducing-valves for the steam drying and heating contrivances, the main work of the engine being the pumping of the water supplies, and driving the ventilating fan and also a carbon dioxide freezing machine, which provides the supplies of ice and cooled water. The reagents room, the mechanical laboratory, the water analysis room, and the hydrometer room, in which all the instruments in use are standardised, also partly occupy the basement.

The ground floor is allotted to the rooms of the Principal and Deputy Principal, the reference sample laboratory, the Crown contracts department, and the research laboratory. The reference sample laboratory, which is typical of most of the rooms as regards arrangements and fittings, may perhaps be described here. The tops of the working benches are of Honduras mahogany, the cupboards beneath them of oak; the bench reagent shelves of plate-glass on gun-metal brackets padded with indiarubber. Each bench is fitted with a metal filter-pump and manometer, a sink for the reception of dirty waste liquid, and a pipe leading to the reservoir under the basement, for conducting away clean used water. On one of the walls is placed a steam drying oven of copper packed in asbestos and cased in oak, and fitted with plate-glass doors. Orifices at bottom and top allow of the passage through it of a current of air which is first heated by being caused to pass through pipes surrounded by the hot packing. The waste steam escaping from the oven is led through a worm lined with pure tin contained in a drum through which cold water circulates, and the resulting distilled water for ordinary purposes collected in a

large earthenware jar fitted with gauge and overflow-pipe. The water used for this condensation and all similar purposes is returned to the clean water reservoir. Beneath the drying oven stands an oak chest which carries at its top a small copper tank, tin-plated inside, and having a tray-shaped copper top. The heated water formed by the condensation of the steam in the pipes of the drying apparatus falls into this tank, and thus yields a supply of hot distilled water, while the hollow cover serves as a bath for heating at moderate temperatures. The cupboard below is used for drying towels, dusters, &c. The floors of the evaporating cupboards and draught chambers are of slate, the top and sides being of plate-glass in oak frames; the fronts open by sliding upward, and are each connected with a counterpoise by a chain passing over a block. Each chamber is connected with the ventilating system by an air passage in the wall of the room. The evaporating apparatus consists of a copper cone sunk in the slate slab, and fitted at its upper part with a removable porcelain collar. This carries a cover of heat- and acid-resisting material perforated with holes of different sizes for the accommodation of basins and capsules. Two holes are pierced at opposite sides of the cone at slightly different heights, the higher being an inlet for steam. This, condensing, fills the cone to the level of the lower orifice, and the water thence runs to a gauge arranged so as to maintain a constant height of water in the cone. Should it for any reason be found impracticable to employ steam, the copper cone can be directly heated by a Bunsen burner fixed beneath it, any loss by evaporation being automatically replaced from the gauge, which is connected both with the water supply and waste cistern. An oak cupboard with top and front of plate glass contains standard solutions. The burettes are fixed on a brass rack immediately above, and are connected by indiarubber tubes to the bottles containing the standardised reagents; they are filled from the stock supply by siphon, the ingress of CO_2 , &c., being guarded against by bulb-tubes filled with soda-lime.

In addition to the apparatus already described, a counterpart of which is to be found in most of the rooms, the Reference Sample Laboratory is also provided with a hot cupboard with sliding plate-glass doors, and a hot trough for melting and filtering butters and fats—a class of substances largely dealt with in this room.

The Crown Contracts Department has for its work the examination of samples submitted on tender for supplies to various Crown Offices and their comparison with the actual deliveries. Besides the fittings common to all the rooms the one allotted to this work has an automatic gas-blast for long-continued ignitions, muffle gas-furnaces for similar purposes, self-regulating hot-air baths, and a sulphuretted hydrogen cupboard with a ventilating passage entering directly into the boiler flue.

The Research Laboratory is fitted similarly with the addition of a hood for combustion work.

Adjacent to the Principal's room is a Reference Library.

On the first floor are the main laboratory, the tobacco laboratory and incinerating room, and the Superintending Analyst's rooms. The former, the largest room in the building, is used for work connected with the beer and spirit duty samples. Leading out of it are a polarimeter room and a cold-storage room, the latter having walls of some 12 in. thick, containing tanks of cold brine and cased with non-conducting material. The water used in the main laboratory itself for condensing purposes is the cooled supply already mentioned: it is kept at a certain fixed temperature convenient for the work.

The fittings of the tobacco laboratory are in the main similar to those of the other rooms, with the exception that extra accommodation for microscopical work is provided. In the incinerating room there is a new device for the first ignition of the samples. This consists of a compact arrangement of small Bunsen burners, the height of the flame of each being independently regulated from

the front. The platinum vessels used are oblong in shape and are supported on a frame of cobalted iron. This material is also used for the inner shell of the muffle furnaces, which are somewhat broader and flatter than is usual. There are three ovens for moisture estimations. These are of the type in general use in the laboratory, but have in addition packed shelves through which the air current passes, entering and leaving them at opposite ends so as to ensure complete circulation in every part of the drying space. As, also, it is necessary for the temperature of these ovens to be maintained throughout the night, they are heated by steam supplied by a self-filling and self-regulating gas-boiler.

Two small staircases lead upwards from the first floor; the one to the photographic dark room, the other to the museum, to the room in which instruction in chemical matters of interest from the Revenue point of view is given to the Supervisors of Excise, and to the typists' and copyists' room. Cloak-rooms on each floor, and a goods lift fill the remaining available space.

THE DETERMINATION OF UNSAAPONIFIABLE OIL IN GREASES WITH A LIME BASE.

By HENRY BAILEY, F.C.S.

THE determination of unsaponifiable oil in greases whose base is an insoluble soap, by the usual method of complete saponification with caustic alkali and extracting the unsaponified oil from the dried soap with a suitable solvent, has, in the hands of the writer, yielded results which at best are unsatisfying, owing to the prolonged drying required to free the soap from water, and the difficulty of completely extracting the unsaponified oil from the pulpy mass.

The following scheme, in which the base is separated from the assay in an early stage of the operation, yields results of maximum accuracy in a minimum of time.

Ten grms. of the sample are weighed accurately into a beaker of about 6 ounces capacity, and boiled with water to which an excess of hydrochloric acid has been added, with constant stirring until the grease is completely decomposed and free from lumps. When decomposition is judged to be complete, the floating oils and fatty acids are separated from the acid solution by filtering through a close-pored filter-paper which has been previously moistened with water, and washed with boiling water until the washings are free from hydrochloric acid. In some cases the filtrate may have a slight iridescent appearance, but the quantity of oil sufficient to cause this is so small that it may safely be ignored. The apex of the filter-paper is now punctured, and the liquid oil washed back into the beaker by a very fine jet of boiling water. About 2 grms. of caustic potash in concentrated solution is now added, together with a little alcohol, and the solution heated with constant stirring until saponification is complete, preferably with the addition of a little more alcohol to replace the solution as it evaporates.

Evaporate most of the alcohol, add about 100 c.c. of water, and warm until clear. The solution is now transferred to a separator, any oil adhering to the beaker being washed in by means of a jet of boiling water; cool, when cold add 30 c.c. of methylated ether or petroleum spirit; shake well, and allow to stand. When the line of demarcation between the aqueous and the ethereal solutions is indistinct, the cautious addition of a few drops of alcohol will improve matters.

Draw off the soap solution, and wash the ethereal solution two or three times with water, adding the washings to the soap. Transfer the ethereal solution to a small tared wide-mouthed flask. The soap solution is replaced in the separator, and again shaken with 20 c.c. of ether

to completely free it from an oil that may have escaped extraction, and the washed ethereal solution added to the weighed flask containing the first extraction.

The ether is now evaporated, and the residue of oil dried in an air-bath at a temperature a little over 100° C. until of constant weight.

My thanks are due to Mr. J. L. Wade, of Nine Elm Lane, S.W., in whose laboratory I have experimented with this method.

THE INTERACTION OF AMMONIUM PHOSPHATE AND CORROSIVE SUBLIMATE IN THE LIGHT OF THE IONISATION THEORY OF SOLUTION.

By D. CARNegie and F. BURT.

LET AB , $A'B'$, $A''B''$, and AB'' represent four soluble salts, having as their proximate components the two acid radicles A and A' , and the two basic radicles B and B' .

By means of very careful measurements of physical properties, it can in general be proved that when solutions of AB and $A'B'$ are mixed, partial mutual decomposition of the salts takes place, an equilibrium being established in which all four salts— AB , $A'B'$, $A''B''$, and AB'' —partake.



But the cases are rare where, by purely qualitative examination involving no measurements, one can demonstrate the occurrence of chemical interaction in such homogeneous systems. As one of these rare cases may be cited the interaction of solutions of sodium chloride and copper sulphate. That solution of these salts *do* interact with the production of some copper chloride and sodium sulphate is evident from the colour change from blue to green which accompanies the addition of salt to solution of blue vitriol.

The following facts furnish a new case where interaction in homogeneous aqueous solution can be proved by purely qualitative means—a case well suited for lecture demonstration.

Neither a dilute solution of sodium phosphate (Na_2HPO_4) nor a dilute solution of ammonium oxalate will give a precipitate when added to a dilute solution of mercuric chloride; but a mixture of solutions of sodium phosphate and ammonium oxalate at once interacts with mercuric chloride, giving a dense curdy precipitate. Exactly the same precipitate is produced when ammonium phosphate solution is added to mercuric chloride. Hence it necessarily follows that solutions of sodium phosphate and ammonium oxalate partially interact with the production of sodium oxalate and ammonium phosphate.

One would off-hand expect the precipitate formed by the interaction of ammonium phosphate and mercuric chloride to be a mercuric phosphate,—to be the result of an ordinary double decomposition between the salts. But if this were the case, then sodium phosphate should act in a similar way. As we have already stated, this is not the case.

As a matter of fact, a quantitative examination showed the precipitate produced by ammonium phosphate to be infusible white precipitate, NH_2HgCl —a substance which is usually obtained as a product of the interaction of mercuric chloride and free ammonia.

Let us now glance at the whole series of these reactions in the light of the new theory of the constitution of salt solutions.

Mercuric chloride undergoes extremely little ionisation when dissolved. This follows at once from the very low electrical conductivity of aqueous solutions of the chloride. Consequently the concentration of the mercuric

ions in HgCl_2Aq is very low—so low, in fact, that the product of the concentration of mercuric ions and the concentration of $\text{C}_2\text{O}_4^{2-}$ ions from soluble oxalate solutions does not exceed the "solubility product" for mercuric oxalate. Hence mercuric oxalate cannot be obtained by interaction of dilute solutions of mercuric chloride and an oxalate.

Similar statements may be made with respect to the absence of any precipitation when solutions of mercuric chloride and any soluble phosphate other than ammonium phosphate are mixed.

When ammonium phosphate is dissolved in water, we get the ions NH_4^+ , NH_4^+ , and HPO_4^{2-} . But phosphoric acid has a very low ionisation tendency; it is, in other words, a very weak acid. Hence the HPO_4^{2-} ions combine with H^+ ions (resulting from the ionisation of water) to form undissociated phosphoric acid. More water and more ammonium phosphate undergo ionisation in the effort to re-establish the equilibrium disturbed by the removal from the sphere of action of H^+ ions and HPO_4^{2-} ions thus brought about. The end result is, that we have in a solution of ammonium phosphate a considerable concentration of free ammonium hydroxide, both in the ionised and un-ionised condition.

Similar statements might be made with regard to the solution of ammonium oxalate, with this difference, however, that oxalic acid being a much stronger—more highly ionised—acid than is phosphoric acid, the ammonium ions never reach a sufficient concentration to cause the visible formation of NH_2HgCl . Some of the NH_4^+ and Hy^- ions do undoubtedly interact in this case, but the product of the concentration of the thus-formed NH_2Hg^+ ions and of the concentration of Cl^- ions never reaches the solubility product for NH_2HgCl .

We found that infusible white precipitate is soluble in excess of $(\text{NH}_4)_2\text{HPO}_4\text{Aq}$, though it is insoluble in excess of either NH_4OHAq or $\text{Na}_2\text{HPO}_4\text{Aq}$. In a solution of ammonium phosphate there must be more free phosphoric acid than in a solution of sodium phosphate of about equal molecular concentration. For ammonia, unlike soda, being a very weak base, the OH^- ions of the water of solution are removed from the sphere of action as undissociated NH_4OH molecules. Hence the concentration of H^+ ions must form by far the more considerable factor in the solubility product for the water which holds the ammonium phosphate in solution. But this means the existence in ammonium phosphate solutions of a comparatively large amount of phosphoric acid.

We therefore attributed the solvent action of solution of ammonium phosphate on white precipitate to the phosphoric acid which the former contains. This being so, white precipitate should dissolve readily in concentrated solutions of phosphoric acid. We found this to be the case, and the beautiful crystals which separate from the phosphoric acid solution are now under investigation.

As the concentration of aqueous salt-solutions rises, the ionisation becomes absolutely less, but the concentration of the ions becomes greater. And as chemical interaction is a function, not of the absolute amounts of the reactions but of their concentrations, we concluded that it might be possible to prepare a mercuric phosphate by precipitation from saturated solutions of mercuric chloride and ammonium phosphate. The dark red precipitate which forms when such solutions are mixed is now undergoing examination. All that can be said at present about it is, that it contains a mercuric phosphate.

Quite apart from the possible establishment of the existence of a new, and probably quite worthless, phosphate of mercury, this precipitate demands careful examination. For if, after long washing, it still be found to contain chlorine, it is clear that in this case (and perhaps in others also) another explanation of its presence lies to hand than the customary one given in such cases, viz., co-precipitation by adsorption.

Clifton College.

LABORATORY EXPERIMENTS.

By J. THOMPSON.

ONE of the simplest experiments to be performed by students of elementary chemistry is the determination of the combining proportion of magnesium and oxygen.

As the magnesium is generally burnt in a limited supply of air, the products of combustion are grey. If a fairly large quantity of this grey substance be exposed to the air for a short time, it will be found to give off the characteristic odour of ammonia.

The grey substance is a mixture of the oxide and the nitride of magnesium. Water vapour acts upon the latter, with the production of ammonia and magnesium oxide.

Platinum and tin, in the proportion of 10 of the former to 1 of the latter (by weight), form a very brittle alloy, giving off a very large quantity of heat. The combination takes place at a red heat, and the globule formed quickly becomes nearly white hot.

SEPARATIONS WITH ALKALINE ACETATES.

By HARRY BREARLEY.

(Continued from p. 167).

IV.—Chromium from Iron.

THAT in ordinary acetate precipitations chromium, if present as a chromic salt, would go down with the iron, is accepted without question. Even with minimum acetate and 10 c.c. of free acetic acid the chromium is almost completely precipitated with the iron; and yet the hydrate, as well as the acetate, of chromium is easily soluble in dilute acetic acid.

There are two phases of the acetate precipitation which, it was thought, might give at least a partial separation. These are—(a) when no free acid is present and barely enough acetate to precipitate the iron, and (b) when large volumes of acetic acid are used with minimum acetate.

The first case was tested by neutralising a mixture of ferric and chromic chlorides, diluting, and heating. At 90° C. there was no sign of turbidity; near the boiling-point the turbidity formed and deepened as the boiling was prolonged, until nearly all the iron was precipitated. Only traces of chromium were to be found in the filtrate. The mixture, as in succeeding cases, contained 1 grm. of iron and 0.1 grm. of chromium. The second case was tested by preparing solutions—both T.H. and N.H.—and precipitating in presence of 30, 50, 70, 100 c.c. acetic acid. The percentage recovery of chromium from the filtrate ranged from 3.75 to 77.5. With other degrees of dilution these results might be improved. With larger amounts of acid, if an inference from the general trend is admissible, a perfect separation might be approximated. But, however possible the separation might thus become, there are inherent difficulties which would prevent the operation ever becoming practicable.

The chief drawback, apart from the expense of the reagents, is the colour of the filtrate. This with the better separations was a deep reddish purple—so deep that in a half-litre flask it was almost opaque. It will readily be seen that on this account the precipitate can only be distinguished from the filtrate by careful examination, and so faulty filtration would often go undetected. Nor would faulty filtration be an unlikely occurrence, because the iron precipitate is so finely divided that no paper I have seen and only a carefully made asbestos filter can completely retain it. The desire to estimate chromium gravimetrically is the chief reason for seeking its separation from iron. In this case that operation would be greatly complicated by the passive nature of chromium acetates formed in

this manner (see CHEMICAL NEWS, xlviii., 114, Reinitzer; also "Bowman's Practical Chemistry," 1878 edition).

The results quoted above were obtained by oxidising with permanganate and titrating with ferrous-ammonium sulphate and bichromate.

The separation of chromium and iron, after oxidising the former to chromic acid, is attributed to Gibbs and is well known. Enquiries lead one to conclude that it is rarely used in steel works' laboratories, and it is rarely seen in text-books. The only recent instance that I know of, having made no special search, in published methods of iron and steel analysis is to be found in CHEMICAL NEWS, lxxvii., 307 (Parry and Morgan). However, it still holds a place in "Select Methods," and thence one might infer that it was at least approximately accurate. I do not mind confessing that I was disappointed to find the method so unsatisfactory, until I reflected that it was no part of my endeavour to satisfy expectations. The avowed principle of the reaction is indeed a prepossessing one. The method, briefly described, is to neutralise, add acetate, oxidise with bromine or chlorine, and boil, whence the iron is precipitated, and the excess of oxidant eliminated simultaneously.

Naturally, the usual amount of acetate, hydrate, and acid were first tried. The tests throughout were made on mixtures of an acidified solution of ferric chloride and potassium chromate (1 grm. Fe, 0.1 grm. Cr). The first few separations were consistently very bad ones (percentage recovery, 26 to 27). It was thought that the presence of a strong oxidant throughout the precipitation was the condition wanting; considerable bromine was therefore added to a mixture, but the result showed no material improvement. Several repetitions of the first experiment emphasized the surprisingly low percentage recovery and drew attention to the unusually low turbidity temperature—50° to 60° C., instead of the expected 80° to 90°. Corresponding results were obtained when the chromate was replaced by bichromate, or the mixture by a sample of chromium steel.

The low turbidity temperature suggested that something in the solution was acting along with the acetate as a precipitant, and that this something could be made to act alone under suitable conditions. The mixture of iron and chromate, therefore, was neutralised, &c., as usual, except that no acetate whatever was added. The solution became turbid at 80° C., and the precipitate and filtrate had much the usual appearance. Less chromium was recovered than in any previous experiment. In a precisely similar experiment, except that the possible formation of acetates was avoided by replacing the 10 c.c. acetic acid by 10 c.c. normal hydrochloric acid, the turbidity appeared at 87° C., and the chromium in the filtrate was less than before. Attempts were made to completely precipitate the chromate by having present more ferric chloride than could be precipitated at boiling heat, and then adding dilute acetate until the precipitation was just accomplished. An impression that the chromate was much the more energetic precipitant of the two suggested this course; it was not successful.

It may be desirable to do more than merely record these observations; here and now, however, it would interfere too much with the purpose of these papers. But it is neither premature nor out of place to suggest that the deficient recovery and the allied low turbidity temperatures are due to a reaction between the chromic acid and the ferric chloride, in which an insoluble chromic compound is formed. "Watts' Dictionary" notices two ferric chromates—a basic salt, $\text{Fe}_2\text{CrO}_4 \cdot 2\text{Fe}_2\text{O}_3$, formed by adding with K_2CrO_4 aq. on iron-alum solutions; and an acid salt, $\text{Fe}_2\text{CrO}_4 \cdot \text{CrO}_3$, said to be formed by digesting CrO_3 aq. with $\text{Fe}_2\text{O}_6\text{H}_6$. If the said compound is either, the probability is that it is the former; anyway, it is a compound of iron with chromic acid, because if the total precipitate be dissolved in dilute hydrochloric acid, and the chromium estimated by Penny's process, as much is

found as makes, with that obtained from the filtrate, an amount exactly equal to that introduced.

Such a method as the preceding can by no stretch of favour be classed as a quantitative one. It is capable of better behaviour surely, else how came it to move amongst "select" methods. It may be noticed on re-reading the instructions that an excess of acetate is recommended. Whether this is an express statement or the customary formulae for acetate precipitations it is difficult to say, but the word "excess" is so important that it should have been printed in italics and then qualified by "very large." I cannot resist the opportunity of repeating that in the separation of nickel and cobalt from iron this course has been adopted. The same eminent metallurgical chemist dismisses the separation of iron and chromic acid with "an excess of sodium acetate" in ordinary type.* This point strongly emphasizes the need of scrutinising methods before adopting them, and the shallowness of the economy which imagines that laboratory operators are dead losses if not producing long rows of results.

It must be admitted that with chromium steels containing only from several tenths to 2 or 3 per cent much better results are possible than I obtained with the previously-mentioned proportions, else such a deficiency could not have gone undetected in the most ill-conditioned laboratory. Where the method has been tested on armour plate borings, with strict adherence to instructions, I am informed that some 50 to 70 per cent of the contained chromium has been estimated. This may readily be explained. The less the percentage of chromium, the greater the proportion of acetate to chromate in the oxidised solution, and the greater the proportion of acetate to chromate (?) precipitated. If this explanation be true, it should happen that all else being equal, the greater volume of acetate should accompany the greater percentage recovery. In Table XIV. this is shown to be so.

TABLE XIV.

Volume of strong acetate. C.c.	Per cent recovery with—	
	Ammonia acetate.	Soda acetate.
10	43.6	50.5
50	47.2	70.0
100	72.0	—
200	81.5	—

The amounts of iron and chromium are as before. The solutions contained total hydrate and 10 c.c. acetic acid. The acetates previously used are too dilute to use in this case, within the limit of our standard volume (1000 c.c.). They were therefore made about 13 times as strong as usual, e.g., the ammonium acetate is approximately that made by neutralising 33 per cent acetic acid with 880 ammonia.

The effect of increasing volumes of acetic acid is to give higher results, but only so in a slight degree.

The foregoing may help to explain why the acetate separation of iron and chromium has fallen into disuse. With such modification as we are now able to make, it is scarcely worth while to formulate the method anew. If there are ever circumstances which make the method a desirable one, well. For determining chromium in iron and steel such methods as Galbraith's, or Stead's modification of it, are all that can be desired, and if a gravimetric method were needed, the separation in question is almost as troublesome and less accurate than fusion with alkalis.

After oxidising the chromium, the acetate is frequently replaced by a pure or carbonated alkali. A few tests rapidly made tend to show that in such cases a similar error—though smaller—is introduced, and may, similarly, be more and more nearly eliminated by adding increasing

* It is only fair to say that the author of these instructions says at the same time "a satisfactory method for the determination of chromium has yet to be devised."

quantities of alkalis. It may be noteworthy, too, that aluminium and chromium are separated under precisely the same conditions as iron and chromium (see "Fresenius," 7th edition, page 428), and a corresponding basic salt, $\text{Al}_2\text{CrO}_4 \cdot 2\text{Al}_2\text{O}_3 \cdot 21\text{H}_2\text{O}$, is said to be formed in a like manner. This makes it desirable to examine the separation of these two elements. Quite casually, too, I learn from an abstract in the *Journ. Chem. Soc.*, that Marchal and Wiernik (*Zeit. Angew. Chem.*, 1891, 511-513) claim to have obtained perfect separations of iron and chromium by oxidising the chromic salt with freshly-precipitated manganese dioxide. Thus it appears that there are a number of methods over which suspicion is cast. If I may reserve this study to myself, it shall not be neglected when "Separations with Alkaline Acetates" have been completed. There is evidence that the use of an alkaline chromate will effect separations which with alkaline acetates are impossible.

(To be continued).

SOME NOTES ON CONCENTRATED SOLUTIONS OF LITHIUM AND OTHER SALTS.*

By JOHN WADDELL, B.A., D.Sc., Ph.D.

In an article published nearly two years ago in the *CHEMICAL NEWS* (lxvii., p. 201), I gave a record of experiments on the relative amounts of water absorbed by the molecular weights of lithium nitrate and calcium nitrate when they were enclosed in the same bottle along with a limited quantity of water. Each of the salts and the water were in tubes like small test-tubes, the quantity of water being small, so that the solutions of the lithium and calcium salts were concentrated. It was found that the lithium nitrate absorbed more water-vapour than the amount calculated on the assumption that lithium nitrate and calcium nitrate are dissociated equally; the indication being, that while neither of the salts is completely dissociated in concentrated solutions, lithium nitrate is more largely dissociated than calcium nitrate. Lithium nitrate was also found to be dissociated to a greater extent than potassium, strontium, or barium, nitrate. It was mainly with a view to seeing whether lithium chloride and sulphate would show similarly greater dissociation than other chlorides and sulphates that the few experiments described in these notes were undertaken.

The experiments are not all complete, the weight of the tubes not being in every case constant; but they have been carried on long enough to make the general conclusions pretty certain, and the results are therefore given without longer delay.

Lithium, strontium, barium, and potassium chlorides were investigated with the results given below:—

Table of Quantities of Water in Grms. taken up by the Molecular Weight (in M.grms.) of the Chlorides.

1 LiCl ..	0.709	0.580	0.597	0.401
1 BaCl ₂ ..	0.966	0.807	—	—
1 SrCl ₂ ..	1.021	—	—	0.604
1 KCl ..	—	—	0.513	0.323

The figures in the last column show that while potassium chloride is not so largely dissociated as lithium chloride, strontium chloride is dissociated to a greater extent. The only alternative to this last conclusion is that both the lithium chloride and the strontium chloride are completely dissociated—a conclusion which is not only *a priori* unlikely, but is negated by the results given in the first column, because there it is seen that the water

absorbed by the lithium chloride is more than two-thirds as much as that absorbed by the strontium chloride, which would seem to prove that the rate of dissociation of the former salt is increasing as compared with the latter. If two-thirds of the lithium chloride and three-fourths of the strontium chloride were dissociated, the number of ions in the one case would be just two-thirds of that in the other; or the same result would follow if one-third of the lithium chloride and one-half of the strontium chloride were dissociated. These are two out of an indefinite number of solutions of the problem, so that it is evident that the experiment does not give sufficient data for determining to what extent the various salts are dissociated.

The amount of water absorbed by the barium chloride is not far short of that absorbed by the strontium chloride, so that it too is probably more dissociated than the lithium chloride.

In the case of the sulphates, lithium was compared with magnesium and zinc. The last two were weighed out in the form of $\text{MgSO}_4 \cdot \text{H}_2\text{O}$ and $\text{ZnSO}_4 \cdot \text{H}_2\text{O}$, that this was their composition being proved by precipitating a portion of the salts with barium chloride.

As the tubes have not attained constant weight, the last two weighings are given, so that it may be seen which tubes were gaining weight and which losing, and at what relative rate.

Table of Quantities of Water in Grms. taken up by the Molecular Weight (in M.grms. of the Sulphates). Those marked + were increasing, those marked — were decreasing.

1 Li ₂ SO ₄ ..	1.108	1.114+	1.790	1.797+
1 MgSO ₄ ..	0.693	0.687—	1.078	0.067—
1 ZnSO ₄ ..	0.620	0.616—	1.070	1.053—
1 Li ₂ SO ₄ ..	—	—	1.924	1.925

The magnesium sulphate is slightly, but only slightly, more absorbent than the zinc sulphate. That the difference of absorption is not large may be seen by noticing that where the magnesium and zinc salts had nearly equal quantities of water, the zinc sulphate was losing more rapidly than the magnesium sulphate; but when the zinc sulphate had only six-sevenths as much water as the magnesium sulphate, the zinc sulphate lost more slowly. The lithium sulphate was far more absorbent than the other two, and plainly—if dissociation into ions is indicated by absorption—the lithium salt is considerably more dissociated than the others.

There were also some further experiments with nitrates, and, in order to show in what direction the action was going in the cases in which the condition was not constant, or so nearly so that the difference in six or seven days was not noticeable, a plus or minus sign is given, with the increase or decrease in the time mentioned.

Table of Quantities of Water in Grms. taken up by the Molecular Weight (in M.grms.) of the Nitrates.

1 KNO ₃ ..	0.402	0.683	1.233+3	1.635-4	3.275
1 LiNO ₃ ..	0.647	0.969	1.611+4	1.968	—
1 NaNO ₃ ..	—	—	1.649	2.218-1	—
1 Ca(NO ₃) ₂ ..	—	—	1.933+2	2.081	—
1 Sr(NO ₃) ₂ ..	—	—	—	2.045	—
1 Ba(NO ₃) ₂ ..	—	—	—	—	—
1 AgNO ₃ ..	—	—	—	—	3.300

The most noticeable feature in this table is that, though potassium nitrate is not so absorbent, and therefore presumably not so much dissociated as lithium nitrate, sodium nitrate, in both cases observed, has taken up more water. Strontium and calcium are not far apart when the dilution is that given above; the difference in more concentrated solutions, as I showed in my former paper, is greater. The barium nitrate weighings are not given, because in the first case water had been added in

* Read before the British Association (Section B), Toronto Meeting, 1897.

large quantity, and all of the gain in the other tubes came from it; in the second case the tube contained crystals.

In the comparatively dilute solutions of potassium and silver nitrate, the absorption was practically equal. Along with these solutions two tubes containing mercurous nitrate were introduced, in order if possible to see whether the formula $\text{Hg}_2(\text{NO}_3)_2$ or HgNO_3 would correspond best with the result obtained, but the absorption of vapour by the salts was so slow that they were not brought into solution, and so nothing could be determined regarding the formula.

I described, in the former paper, some experiments with potassium chloride, bromide, and iodide, which proved that the invaporation of these salts, provided there is enough water present to bring them all into solution, is approximately equal. I have since compared potassium chloride and potassium nitrate, and, as the table shows, the chloride is very considerably more absorbent.

Table of Quantity of Water in Grms. taken up by the Molecular Weight (in M. grms.).

1 KCl	0.641	0.902
1 KNO ₃	0.482	0.780

The dissimilarity between the chloride and nitrate is all the more remarkable because they agree much more nearly as to solubility than the chloride does with the bromide and iodide. If the first table containing the chlorides, and the table containing the nitrates, be referred to, it will be seen that lithium and potassium chloride differ less in the amount of water absorbed (or, in other words, have more nearly the same vapour pressure for the same strength of solution) than lithium and potassium nitrate. Indeed, though with the concentrated solution that I had, the lithium chloride is more dissociated than the potassium chloride; the table of electrical conductivities given by Kohlrausch indicates that if the dilution were twice as great the potassium chloride would be the more dissociated.

The conclusion to be arrived at from my experiments is, that there is no special peculiarity belonging to lithium salts as regards invaporation power, there being some chlorides and some nitrates that absorb more water per molecule.

The behaviour of salts in the concentrated solutions is so haphazard that there is little more to be done with them, and I should not consider the investigation worth pursuing further in this direction. The method is, however, adapted for comparing the vapour pressure of different solutions. For example, if it is desired to show the student that molecular weights of such salts as potassium chloride and potassium iodide, having equal quantities of water, have the same vapour pressure, it would merely be necessary for him to make two such solutions, and arrange three experiments in one of which the solutions are used as prepared above, a second in which the potassium *chloride* is made slightly more dilute, and a third in which the potassium *iodide* is made slightly more dilute. These three bottles with the enclosed tubes may be set aside for a few days, and then on weighing there will be found to be no change in the tubes in the first bottle; in the second, the tube containing potassium chloride would be found to be losing weight and the iodide gaining; while in the third, the reverse action would be seen to be going on.

In this way the well-known law of vapour pressures could be illustrated without any elaborate apparatus. Moreover, if a number of experiments are to be performed, it is easier to set them up and allow them to stand for a few days than to go on with a series of determinations of vapour pressure in an apparatus which must be carefully cleaned and dried each time.

Why my experiments took so long was because I was working at the invaporation from the beginning, and did not make use of solutions made up to a suitable strength, my purpose being different from the one of which I am

now speaking. As I have stated, there is not much regularity in the concentrated solutions, no law having been found by which it would be possible to predict which of two concentrated solutions would have the greater vapour pressure; but this method of comparing vapour pressures may sometimes be found valuable.

Kingston, Canada.

THE TITRATION OF SODIUM THIOSULPHATE WITH IODIC ACID.*

By CLAUDE F. WALKER.

THIS investigation was undertaken to determine the nature and limitations of the reaction between iodic acid and thiosulphuric acid, and to show the expediency of employing iodic acid in standard solution for the direct titration of sodium thiosulphate. Riegler states that iodic acid is readily obtained pure, and that a standard solution can be kept a long time unaltered. He further states that when a solution of sodium thiosulphate is titrated with iodic acid the reaction takes place according to the equation—



Under which circumstances no free iodine will be evolved until all the sodium thiosulphate has been oxidised to tetrathionate. The first drop of iodic acid in excess, however, will react with the sodium iodide that has been formed and liberate iodine, as shown by the equation $5\text{NaI} + 6\text{HIO}_3 = 5\text{NaIO}_3 + 3\text{H}_2\text{O} + 3\text{I}_2$, thus furnishing an accurate means for determining the end of the reaction.

A careful repetition of Riegler's work shows that his conclusions are to a great extent erroneous. Thus, "chemically pure" iodic acid is very likely to contain too much iodine, due probably to the presence of the anhydride. Riegler's proposed method of titration depends on two different reactions, and these must be definite, complete, and non-reversible under the conditions of the analysis. Thus one molecule out of every six of iodic acid should be reduced by six molecules of thiosulphate, forming a neutral mixture of iodide and iodate, when it might be expected that iodine would be liberated by the first trace of iodic acid in excess. There was striking evidence, however, of some obscure reaction of the thiosulphate, which influences the reduction of the iodic acid, so as to make it impossible to calculate analyses according to Riegler's reaction. It is not impossible that some third unstable compound of iodine may be formed as an intermediate product and delay the titration of iodine.

It appears, however, that Riegler's proposed process for standardising sodium thiosulphate, is impracticable unless it can be so modified as to do away with a number of sources of error.

The analyses of solutions of iodic acid in this work was invariably performed by adding to the portion of the solution to be analysed an excess of potassium iodide, acidifying with 5 c.c. of dilute (1:3) sulphuric acid, and recovering the liberated iodine by titrating the acid solution with sodium thiosulphate, or by neutralising with an excess of potassium bicarbonate, and titrating the alkaline solution with arsenious acid. In either case one-sixth of the iodine recovered was calculated to iodic acid, according to the equation $5\text{HI} + \text{HIO}_3 = 3\text{I}_2 + 3\text{H}_2\text{O}$.

In the present work it was found convenient to analyse the iodic acid in quantities of about one-tenth of a gram, when the variation in results in the same series is inappreciable.

By blank experiments it was found that one drop of iodine was necessary to bring out the starch blue, so this was uniformly applied in the analytical work. Two dif-

* Abridged from the *American Journal of Science*, vol. iv., Sept., 1897.

ferent samples of "chemically pure" iodic acid were used to determine whether its purity is sufficient to admit of its direct application in standard solutions. Quantities of both these were dried over sulphuric acid to constant weight. A third sample was prepared by dissolving the purest obtainable iodic anhydride in water and evaporating at ordinary temperature.

The resulting crystalline mass was dried over sulphuric acid for a week, until ceasing to lose weight; it was presumed to consist of the pure normal acid. Two presumably decinormal solutions of each of the first two samples, and one such solution of the third sample of iodic acid, were made by dissolving 17.585 grms. in 1 litre of water at 15° C. Analyses of these solutions gave the following results:—

Solution.	Sample used.	HIO ₃ taken. Grm.	HIO ₃ found. Grm.	Error. Grm.
I.	A	0.1055	0.1066	0.0011+
II.	A	0.1055	0.1062	0.0007+
III.	B	0.1055	0.1065	0.0010+
IV.	B	0.1055	0.1073	0.0018+
V.	C	0.1055	0.1053	0.0002—

These results, which are averaged from a large number of determinations, show that while the deviation from the theoretical strength of the solution in the case of the acid prepared from anhydride is hardly appreciable, the solutions made from the purchased product contain a very appreciable amount of iodine in excess of the theoretical quantity.

To determine whether a solution of iodic acid, once prepared and standardised, will retain its strength for a long time, two such solutions were kept for four months (in the dark) and then again analysed. The results (averages of several determinations) given below substantiate Riegler's observation that a solution of iodic acid will remain of constant strength.

Constancy of Strength of Iodic Acid Solutions.

Iodic acid solution.	First analysis. HIO ₃ found. Grm.	Second analysis (after four months). HIO ₃ found. Grm.	Variation. Grm.
I.	0.1073	0.1072	0.0001—
II.	0.1049	0.1046	0.0003—

A series of analyses made by oxidising the sodium thiosulphate to sulphate and precipitating and weighing as barium sulphate, gave results identical with those obtained with iodine; proving that all the sulphur present was in the form of thiosulphate.

According to Riegler's equation, sodium thiosulphate and iodic acid react molecule for molecule, and solutions of them should therefore require for their mutual saturation volumes inversely proportional to their concentration. It was found, however, that when a one-twentieth normal solution of sodium thiosulphate was titrated in the presence of starch with an approximately decinormal solution of iodic acid, a distinctly blue colour was produced long before the theoretical amount of iodine had been added. It was further noticed that the end-point of the reaction was far from distinct; a faint tint of blue at first being visible, then suddenly becoming deeper, and immediately reappearing when bleached with sodium thiosulphate. It was found, however, that the addition of a considerable quantity of potassium iodide to the solution, either before or during the titration, had the marked effect of making the reaction sharp and distinct, entirely preventing the "after separation" of iodine, and at the same time postponing the appearance of the starch-blue until a quantity of iodic acid had been added considerably in excess of the theoretical. The experiments were executed with entirely different reagents, and under varied conditions of concentration, the results in every case confirming those already observed.

For the purpose of more particularly investigating this subject, there were prepared and standardised an approximately decinormal solution of sodium thiosulphate and an approximately one-fiftieth normal solution of iodic acid. Measured portions of the sodium thiosulphate solution were titrated with the iodic acid in presence of starch under varying conditions of mass, time, and dilution.

To determine the variability of the end-point of the reaction, a series of experiments was made. Measured amounts of the thiosulphate solution were drawn into an Erlenmeyer beaker, 5 c.c. of starch were added, and the iodic acid slowly dropped in, with constant agitation, until the first blue tint appeared. The results obtained were as follows:—

Variation of the End-reaction between N/10 Sodium Thiosulphate and N/50 Iodic Acid in the Absence of Potassium Iodide.

	Na ₂ S ₂ O ₃ taken. C.c.	HIO ₃ added. C.c.	Mean value. C.c.	Variation. C.c.
1.	6	28.13	28.32	0.19—
2.	6	27.79		0.53—
3.	6	28.03		0.29—
4.	6	28.32		0.00
5.	6	28.32		0.00
6.	6	28.71		0.39+
7.	6	28.83		0.51+
8.	6	28.43		0.11+
9.	4	18.94	18.68	0.26+
10.	4	18.67		0.01—
11.	4	18.50		0.18—
12.	4	18.60		0.08—

These experiments indicate that the constancy of the end of the reaction in different titrations of equal volumes of the same solution depends to a certain degree on the volume of sodium thiosulphate taken. The probable error which these irregularities would introduce in any series of practical analyses by this method is obviously greater than can ordinarily be permitted in iodometric work.

The experiments in the following table were performed in exactly the same manner as those in the last series, except that 2 grms. of potassium iodide were added to the sodium thiosulphate before titrating.

Variation of the End Reaction between N/10 Sodium Thiosulphate and N/50 Iodic Acid in the Presence of Potassium Iodide.

	Na ₂ S ₂ O ₃ taken. C.c.	HIO ₃ added. C.c.	Mean value. C.c.	Variation. C.c.
1.	6	32.53	32.48	0.05+
2.	6	32.45		0.03—
3.	6	32.67		0.19+
4.	6	32.37		0.11—
5.	6	32.36		0.12—
6.	6	32.50		0.02+
7.	4	22.30		0.11+
8.	4	21.98	22.19	0.21—
9.	4	22.17		0.02—
10.	4	22.30		0.11+

These experiments show that in the presence of iodide of potassium the end reaction is practically independent of the amount taken for analysis. It is therefore evident, from studying the above figures, that the presence of potassium iodide in the sodium thiosulphate to be titrated will bring the variation of the formation of the reading tint within permissible limits.

Another series of experiments was made to determine the nature and effect of the "after colouration" observed to take place when a solution of sodium thiosulphate, free from potassium iodide, was titrated with iodic acid to blue colouration and then bleached with sodium thiosulphate. The results are given below:—

*Effect of Dilution and Lapse of Time on the
"After Colouration."*

Na ₂ S ₂ O ₃ taken. C.c.	HIO ₃ added. C.c.	Na ₂ S ₂ O ₃ added. C.c.						Vol. C.c.
		15 mins.	45 mins.	1 hr. 45 mins.	2 hrs. 45 mins.	20 hrs.	Total.	
1.	6	27'68	0'25	0'13	0'08	0'00	0'03	0'49
2.	6	27'70	0'20	0'10	0'03	0'03	0'03	0'39
3.	6	28'17	0'16	0'10	0'03	0'01	0'00	0'30
4.	6	27'03	0'60	0'26	0'09	0'03	0'00	0'98
5.	6	27'60	0'93	0'28	0'06	0'04	0'04	1'35
6.	6	28'60	1'34	0'46	0'17	0'03	0'14	2'14
7.	6	28'85	1'20	0'50	0'28	0'06	0'27	2'31
8.	6	31'63	1'46	0'74	0'10	0'21	0'23	2'74
9.	6	29'90	1'04	0'60	0'23	0'15	0'46	2'48
10.	6	36'09	1'60	1'23	0'63	0'34	0'18	3'98
11.	6	37'59	1'65	1'33	0'72	0'27	0'10	4'07
12.	6	37'23	1'92	1'05	0'64	0'33	*	300

* No observation.

In the experiments with small volumes, the evolution of iodine in any quantity ceased after two or three hours, though the solution would become coloured as often as it was bleached for a number of days. The traces of iodine thus set free were, however, only equivalent to one or two drops of sodium thiosulphate, but the larger volumes continued to liberate iodine in abundance for a very long time. The amount of iodine thus liberated after the first colouration evidently varies with the amount of iodic acid required for the titration; both of these quantities increase at a regular rate with the volume of the solution.

To try with what accuracy the reaction between sodium thiosulphate and iodic acid may be applied to the direct estimation of one of these substances by the other, the averaged results of a large number of titrations are here given. The operations were conducted as directed by Riegler, equal volumes of standard thiosulphate being titrated with iodic acid of known strength in the presence of starch, and under different conditions of time, dilution, and mass, the volume of iodic acid required being in each case compared with the volume theoretically required by Riegler's equation.

*Titration of N/10 Sodium Thiosulphate with N/50
Iodic Acid.*

Iodine Atom.						
Na ₂ S ₂ O ₃ taken.	HIO ₃ added.	HIO ₃ required by theory.	Error.	Error.	Present.	Vol.
C.c.	C.c.	C.c.	C.c.	Per cent.	Grm.	C.c.
1. 4	18'68	20'32	1'64-	8'0-	—	50
2. 6	28'32	30'48	2'16-	7'0-	—	50
3. 6	27'32	30'48	3'16-	7'0-	—	150
4. 6	28'73	30'48	1'75-	6'0-	—	200
5. 6	30'77	30'48	0'29+	0'01+	—	250
6. 6	36'97	30'48	6'49+	21'0+	—	300
7. 6	27'46	30'48	3'02-	10'0-	—	50
8. 6	26'15	30'48	4'33-	14'0-	—	150
9. 6 †	26'50	30'48	3'98-	13'0-	—	200
10. 6	27'16	30'48	3'32-	10'0-	—	250
11. 6	32'93	30'48	2'45+	8'0+	—	300
12. 4	22'19	20'32	1'87+	9'0+	0'2	50
13. 6	32'48	30'48	2'00+	7'0+	0'2	50

* HIO₃ added until first blue colour.

† Calculated by subtracting from the amount of iodic acid originally titrated the volume of thiosulphate required to bleach the solution after standing twenty hours.

These results show plainly that the amount of iodic acid required to decompose a given amount of sodium thiosulphate may be considerably above or below that required by Riegler's equation. Thus, with small volumes, and in the absence of potassium iodide, the thiosulphate is destroyed, and the separation of iodine commences when only 93 per cent of the theoretical amount of acid has been titrated. At higher dilutions the action is retarded, so that at 250 c.c. very nearly the theoretical amount of acid is required to produce the first blue colour, and at 300 c.c. an excess of 21 per cent over the theoretical

amount must be added, and it appears that for all volumes below 300 c.c. the original thiosulphate is destroyed when about 90 per cent of the theoretical amount of iodic acid has been added. Potassium iodide retards the action, so that at small volumes an excess of about 8 per cent of iodic acid must be added to completely destroy the thiosulphate and commence the separation of iodine. It is obvious from the preceding experiments that the reaction between iodic acid and sodium thiosulphate is so indefinite in its nature, and so dependent for its completeness on conditions of time, dilution, and mass, that its direct application as a means of standardising solutions must remain impracticable.

ACETYLENE GAS.

THE Explosives Department of the Home Office has recently had under consideration the question of the restrictions to be applied to the manufacture and keeping of acetylene gas, and has conducted various experiments with the object of gaining information on this matter. The results show conclusively that acetylene gas *per se*, when under a pressure of something less than two atmospheres, is violently explosive; whereas at a pressure of less than one and a half atmospheres it appears to be reasonably free from liability to explosion, provided it is not admixed with oxygen or atmospheric air.

For commercial and practical purposes it is considered sufficient to allow a pressure of 20 inches of water above that of the atmosphere (*i.e.*, roughly, about one and one-twentieth atmospheres), and it is accordingly proposed to draw the safety line at this point, and to declare acetylene when subject to a higher pressure to be an "Explosive" within the meaning of the Explosives Act, 1875.

In France and Germany the authorities have fixed the limit of danger at one and a half and one and one-tenth atmospheres respectively, and have imposed prohibitions or restrictions on the keeping or manufacture of the gas when it is at a higher pressure.

Whitehall, October 5, 1897.

NOTICES OF BOOKS.

An Electrical Method of Determining the Moisture Content of Arable Soils. By M. WHITNEY, D. GARDNER, and L. J. BRIGGS. Washington: Government Printing Office. 1897.

THE only method in general use for determining the moisture in soils is the very simple one of taking a sample of the soil from the field, at any desired depth, and drying it at 100° C.; but from a large number of observations it is found that this method is not accurate to within 2 per cent, plus or minus.

Another method which has been tried is that of burying bricks about 8 inches below the surface, and taking them up and weighing them day by day; this method is obviously of no use whatever. The possibility of using the electrical resistance of soils for the determination of moisture was suggested by the necessity of getting a good "earth" for lightning conductors, &c.

Soils are composed of fragments of various minerals and salts, all more or less soluble in water; even quartz is slightly soluble in water containing carbonic acid, as soil waters usually do, and this dilute solution conducts the electric current. The specific resistance of a solution is the resistance of 1 c.c. of liquid between two parallel electrodes 1 c.m. square and 1 c.m. apart, and the specific conductivity as used in this Bulletin is the reciprocal of the specific resistance.

The Wheatstone Bridge method was used with an alternating current and a telephone in place of a galvanometer. The electrodes finally adopted for field-work consist of carbon plates, each 3 inches long, $\frac{3}{8}$ inch wide, and $\frac{1}{4}$ inch thick, copper-plated at one end, to which a copper wire was soldered; the plates can be placed at any desired depth, but 1 to 2 feet has been found the most convenient; but we are unable to find any definite statement as to how far apart they are placed. The whole apparatus is standardised by comparing the readings with a number of observations taken by the old method, and a table of values is constructed.

An Electrical Method for Determining the Temperature of Soils. By M. WHITNEY and L. J. BRIGGS. Washington: Government Printing Office. 1897.

In perfecting the electrical method of determining the moisture in soils, a compensation cell, having the same electrical temperature coefficient as the soil, is used as one arm of the Wheatstone Bridge, in order to eliminate temperature effects in determining the electrical resistance of soils, and this cell can be very easily used for taking the temperature of the soil.

These cells are constructed of small strips of glass, cemented together with marine glue; their dimensions are 3 inches $\times$ 2 inches $\times$ $\frac{1}{4}$ inch; the electrodes consist of strips of carbon, to which wires are soldered, cemented to the glass. They are then standardised, and the resistance reduced to a common standard, viz., 1000 ohms at 60° F. on being buried, and readings taken with the Wheatstone Bridge; the temperature is arrived at by calculation.

An Electrical Method of Determining the Soluble Salt Contents of Soils. By M. WHITNEY and T. H. MEANS. Washington: Government Printing Office. 1897.

A VERY simple and delicate method has been devised for determining the soluble salt contents of soils, in samples taken from the field. The method consists essentially of mixing a known quantity of a soil with a known proportion of pure water, and determining the specific resistance; then an equal weight of the same soil is mixed with a weak salt solution equal in volume to the pure water added, and the specific resistance again taken; the amount of salt added being accurately known, and also the effect it had on lowering the resistance, it is easy to calculate the quantity of salts originally present in terms of the salt solution used.

A Treatise on Medical and Scientific Radiography. ("Traité de Radiographie Médicale et Scientifique.") By Dr. FOVEAU DE COURMELLES. Pp. 470, with 176 illustrations. Paris: Odeve Doin. 1897.

PROFESSOR RÖNTGEN's discovery, that the X rays emitted by a Crookes tube affected a sensitised photographic plate, is only of recent date, being first made known at the latter end of 1895; but already, in this short space of time, enormous developments have been made in the subject, more especially in connection with surgery, and the subsequent discovery of the phosphorescent screen enables us not only to photograph the internal organs, but practically to see them. A great deal has already been written on this subject, but mostly in the form of papers and articles. The large volume now before us treats of the whole subject from its beginning up to as far as it is at present understood, and the author states that if his book rapidly becomes old-fashioned he will be the more happy, as it will show that we are making further advances in our knowledge of the subject.

In Chapter I. the early experiment of the Abbé Nollet, in 1753, is first mentioned, but we rapidly come to more modern times, when the so-called "black light" of

Moser, and Breguet, the stratified discharge of Crookes, the experiments of Hertz and Lenard, and finally Röntgen's discovery, are described and discussed.

Chapters II. and III. are devoted to the enumeration of the different sources of electrical energy, electrolysis, accumulators, &c.; while in Chapters IV. and V. dynamo-electric and electrostatic machines are dealt with. We next come to currents of high frequency, radiations both invisible and of intense therapeutic action, no longer in a vacuum, but permeating the atmosphere with the greatest ease. A lot of apparatus of special forms, adapted to medical wants, is described in the succeeding chapters, together with the methods of manufacture, pumps, coils, &c.

The form of the bulbs has been varied considerably by different workers, but they may be divided into three groups,—those in which the cathodic rays act directly on the sides of the glass tube itself, those where the cathodic rays are concentrated on some sort of a mirror which reflects them, and those in which the two preceding ones are combined. The first is the most simple, but the second form is more practicable and is the one mostly used, the indirect action being much more intense, and requiring a shorter time to obtain good results.

The radioscope, which we find in Chapter XIV., enables us to see at once certain parts of the interior of human bodies, as well as through other substances, and a very good illustration shows the manner of its application. It is now being extensively used in the French hospitals, not only for examining broken bones or searching for bullets, but also for diagnosing diseases, such as pleurisy, tuberculosis, gout, rheumatism, &c., while the internal deformities in women caused by tight lacing are very easily detected. In fact, it is medical and surgical science that has benefitted principally by the discovery and application of the X rays.

The volume is one of great interest and fascination; it is well printed, on good paper; it has but one fault, one very common to French books—there is no Index.

The Manufacture of Artificial Mineral Waters and other Effervescent Beverages. ("Die Fabrikation der Künstlichen Mineral Wässer und anderer Mousseurnde Getränke.") By Dr. B. HIRSCH and Dr. P. SIEDLER. Third Edition, with 103 figures. Brunswick: Vieweg and Sons. 1897. 8vo., pp. 386.

THE work before us is written not merely in the German language, but most decidedly from a German point of view.

The authors treat, in the first place, of mineral waters in general, of the origin and ingredients of the springs, of the changes to which they are liable, their synthesis and classification. Their classification is complicated, as it is founded both on chemical and medicinal considerations, or on a combination of these two points of view.

Thus we have here alkaline waters, sulphate of soda waters, chalybeate brines, bitter waters, sulphated waters, earthy or calcareous waters, neutral thermæ, and poor in mineral matters.

As a distinct class are mentioned semi-natural mineral springs. Here comes the admission that relatively few springs possess well-marked medicinal properties, but serve merely as a base for the addition of drugs.

There exist also "semi-mineral waters," to which extraneous matter has been added to improve the taste; the principal of these additions is carbonic acid, which is employed both as a gas and as a liquid.

The admittedly artificial mineral waters were mentioned by Pliny, and in modern times were produced by Priestley and Lavoisier, and in 1787 artificial Selzer water was produced on the large scale by Meyer of Stettin, and the manufacture is still flourishing. Some of the artificial waters consist merely of water saturated with carbonic acid, but in other cases soluble salts are

added. The greatest care is—or ought to be—used in the selection of the water. It should be colourless and inodorous, even when warmed and exposing an extensive surface. Nor should it have a sweetish, bitter, chalybeate, inky, saline savour.

It is to be noted that the non-European mineral springs have been overlooked. This is to be regretted. South Africa is exceptionally rich in mineral waters, as was shown in the Indian and Colonial Exhibition. The question arises whether the importation and sale of South African waters should not be promoted in this country, so as to take the place of those from the European Continent, which by vigorous advertising have almost secured a monopoly of the home market.

The Organised Science Series. First Stage: Sound, Light, and Heat. By JOHN DON, M.A., B.Sc. London: W. B. Clive, University Correspondence College Press. Pp. 307.

THIS book is ably and clearly written, and no one need search for errors in its teachings unless anxious to lose his labour. At the same time it is most peculiar, and we may venture to say unhappy. It is examinational to the core, and boasts of the number of Correspondence College students who have "passed" during the last five years. How many of them have made any discovery—in other words, have contributed—to the sum total of human knowledge we are not informed. We may perhaps be pardoned for doubting whether the author considers this the grand object to be aimed at by a University, or is not perhaps fully content with securing for his students exhibitions, scholarships, and "places in honours."

CORRESPONDENCE.

SUPPOSED NEW ELEMENT WITH IRON.

To the Editor of the Chemical News.

SIR,—In last week's number of the *CHEMICAL NEWS* (vol. lxxvi., p. 171) I noticed a letter from Mr. Jones, in which he suggests that the metal I separated from iron might prove to be molybdenum. The properties of the metal do certainly resemble those of molybdenum very closely—a fact of which I have been fully aware. In my first notes on the reactions of the metal I find that "a solution of the metal heated with Na_2HPO_4 and a few drops of HNO_3 produce no precipitate." How I managed to leave out this reaction in my article in the *CHEMICAL NEWS* I really do not know. The metal has been repeatedly tested since then for molybdenum, but without success.

I have during the last week gone over the reactions of the metal, the metal used being specially prepared for the purpose. I could find no traces of molybdenum, nor was I able to find any fault with the reactions of the metal as described in my article in the *CHEMICAL NEWS*.

I have also specially prepared some metal from boiler-dust; it was most carefully tested for molybdenum, but without success.

A small quantity of metal obtained from boiler-dust a considerable time ago yielded a very small quantity of molybdenum, but not sufficient to interfere with the reactions of the metal from which it was separated.

The fact that the metal is precipitated by zinc (the characteristic blue colour produced by the action of zinc on molybdenum being conspicuous by its absence), that it combines with hydrogen (producing a gas from which the metal can easily be deposited), and that it is not precipitated by Na_2HPO_4 and HNO_3 when warmed, led me to believe that the metal was not molybdenum, as these are

certainly not properties of the latter metal as far as I know.—I am, &c.,

GETHEN BOUCHER.

The Laboratory,
North Lonsdale Iron and Steel Company, Lim.,
Ulverston, Lancashire, October 6, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Moniteur Scientifique,
Series 4, Vol. xi., Part 2.

Review of Photography.—A. Granger.—According to recent information the Americans have been for some time using chromo-photography for printing advertisement posters. This has been done by means of three monochromes, and the following solutions have been recommended. For violet—7 c.c. of concentrated solution of cupric chloride, 17 c.c. of water, and 3 c.c. of ammonia; after filtration 3 c.c. of concentrated methyl violet B and 5 c.c. of fuchsine is added. For orange—15 c.c. of concentrated solution of chloride of cobalt, 35 c.c. of water, 25 c.c. of bichromate of ammonium, and 2 c.c. of ammonia. For green—a solution of sulphate of nickel. These solutions are placed in reservoirs of plate glass $\frac{1}{8}$ th of an inch thick.

New Methods of Converting Paranitrodiamino-triphenylmethanes into Fuchsines, or into Bases of the corresponding Fuchsines.—Maurice Prud'homme.—Already noticed.

Contribution to the Study of the Formation of Amylic Alcohol in Commercial Fermentations.—Lucien Gentil.—The presence of amylic alcohol in industrial fermentations has always been observed in more or less quantity, and the question of whether this alcohol was a constant product of alcoholic fermentation, as are succinic acid and glycerin, has been the subject of a good deal of discussion, and the author is led to conclude that it is not formed in a perfectly sterile medium, which is then infected with a pure cultivation of yeast.

Some Reactions of Phospham.—R. Vidal.—When phospham, PN_2H , is heated to a bright or strong red heat in the presence of alkaline carbonates the following reaction takes place:— $\text{PN}_2\text{H} + 2\text{CO}_3\text{R}_2 = \text{PO}_4\text{R}_2\text{H} + 2\text{CNOR}$. That is to say, an alkaline cyanate is formed; the introduction of a reducing agent, such as carbon, into the mixture gives an alkaline cyanide. If iron is used instead of carbon a ferrocyanide is produced, and with sulphur a sulphocyanide. Methyl and ethyl alcohols react on phospham, giving a free secondary amine and the metaphosphate of a primary amine. The reaction of phospham on normal propyl alcohol is more complex, for besides the formation of propylamine we observe a notable quantity of oxide of propyl. With ethylenic glycol the reaction is entirely reversed, and a complete dehydration only is observed; the phospham taking away two molecules of water, forming di-ammonium orthophosphate and setting acetylene at liberty.

Bulletin de la Société d'Encouragement pour l'Industrie Nationale. Series 5, Vol. ii., No. 8, August, 1897.

Report on M. Gosart's Acetylene Lamp.—M. Violle (for the Committee of Economic Arts).—In this lamp the acetylene is generated as is usual by the action of water on carbide of calcium, but its inventor has made use of the action of capillary tubes, whereby the water is brought in contact with the carbide in very minute quan-

ties, and automatically only in the quantity required. By turning off the water tap and the gas outlet tap at the same time the apparatus ceases to work, and no more gas is formed until both taps are turned on again. By turning off the water tap only, the disengagement of gas becomes slower, and gradually stops. If, on the other hand, the gas tap only is turned off, the gas continues to be evolved until the pressure is sufficient to drive back the water in the capillary tubes, which thus act as a safety valve. The production of gas is thus intimately dependent on its consumption.

Journal de Pharmacie et Chimie.
Series 6, vol vi., No. 3.

Decomposition of Iodoform by Light.—G. Fleury.—There is a great diversity of opinion with regard to the action of light on iodoform; the author shows that, as a rule, this decomposing action is of very limited extent, and he thinks that this limit may be attributable to immediate colouring of the liquid to reddish brown by the liberated iodine stopping the violet and ultra-violet rays on the surface, and thus preventing the continuance of the chemical reaction. With the idea of proving the correctness of this theory, some iodoform—dissolved in a mixture of alcohol and ether—was placed in a flask of white glass, in the presence of an excess of finely divided silver. It was then exposed to sunlight, both direct and diffused, and frequently shaken to accelerate the combination of the liberated iodine with the silver. After several days, the liquid no longer became coloured, and on collecting and weighing the mixture of silver and iodide, the amount of iodine resulting from the decomposition was calculated and found to be the exact theoretical amount.

On the Essence of Bitter Fennel.—E. Tardy.—The result of this research shows that the essence of cultivated French bitter fennel contains a dextro-divalent turpentinic carbide, a dextro-tetravalent terpene, an inactive carbide called cymen, fenone, estragol, anethol, anisic aldehyd, anisic acetone, anisic acid, and a crystalline body corresponding to the formula $C_{13}H_{14}O_2$.

Estimations of Commercial Albumen.—P. Carles.—Already noticed.

Colorimetric Reaction of Disulphuric Acid.—E. Barral.

Analysis of an Artificial Roasted Coffee.—F. Coreil.—The sample examined was found to be made by moulding a mixture of flour and husk of wheat, potato, leguminous flour, gum, vegetable refuse, &c., and to contain none of the elementary constituents of true coffee berries.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xvii.-xviii., No. 15.

M. Tanret delivered a discourse on the lamented death of M. Schützenberger, paying tribute to his bold and lofty views, aided by a rare talent of observation and experimentation. Thanks to his numerous researches, of which only a small number could be here mentioned, the name of Schützenberger will live for ever in Science, and will find a place among the foremost of modern chemists.

M. Tanret presented a note, on behalf of M. Sabatier, "On Blue Nitrodisulphonic Acid and some of its Salts."

On Exact Cryoscopy. Correction for Surfusions, and a Means of Recognising a Good Cryoscopic Method.—A. Ponsot.—Amongst the methods used for making the correction for surfusion, that of M. Raoult consists of finding experimentally the points of congelation of the solution A' , A'' , corresponding to degrees of surfusion δ' , δ'' , &c.; a graphic curve gives A . The variation for 1° of surfusion from the point of congelation observed in a solution divided by the lowering is called K . He shows that K is variable with the concen-

tration of a solution, and with the nature of the body dissolved.

On a Colorimetric Reaction of Disulphuric Acid.—E. Barral.

On the Thermic Study of Suberic Acid.—G. Massol.—Suberic acid occurs in the form of brilliant white laminated crystals, melting at 139.5° , and slightly soluble in cold water. It forms suberate of potash easily at the ordinary temperature. The neutral salt dried at 100° is anhydrous, and easily dissolves in water. The acid suberate of potash is very little soluble in water; its heat of solution at about 40° is 5.26 calories, that of the neutral salt being 0.92 calories.

On the Thermic Study of Sebacic Acid.—G. Massol.—The acid salts of potash and soda are but slightly soluble in water; the anhydrous neutral sebate of potash dissolves in absorbing 1.33 calories. The heat of formation of neutral suberate of potash is considerable for an acid of so high a molecular weight, viz., 43.99 calories.

General Considerations on the Normal Di-acids of the Oxalic Series.—G. Massol.—The heats of formation of the neutral anhydrous salts of potash for the six acids the author has studied are as follows:— C_2 , oxalic, $=58.97$ cal.; C_3 , malonic, $=48.57$ cal.; C_4 , succinic, $=46.40$ cal.; C_5 , glutaric, $=44.23$ cal.; C_6 , suberic, $=44.76$ cal.; C_{10} , sebacic, $=43.99$ cal.; 2 molecules of acetic acid $=43.72$ cal. He considers that the quantities of heat which measure the chemical affinity decrease as the molecular weights increase, the heat of formation tends to become low, the reciprocal influence of two carboxyls decreases very rapidly by the introduction of one or two CH_2 intermediaries. The influence of two carboxyls ought to betray itself by the possibility of the formation of internal anhydrides. The acid normal alcohols of the lactic series present analogous facts; for instance, the reciprocal influence of acid oxyhydriles and alcohol is shown by the formation of lactones.

On Acetylmethylheptenone.—Ph. Barbier and G. Leser.—Acetylmethylheptenone soda, treated with iodide of ethyl, gives ethylacetylmethylheptenone, boiling at $133-135^\circ$ under 15 m.m. pressure.

Contribution to our Knowledge of Aliphatic Nitramines.—A. P. N. Franchimont.—All the aliphatic nitramines, acid and neutral, when treated with an acetic solution to which is added either α -naphthylamine, aniline, dimethylaniline, &c., give, with a scrap of zinc, either yellow, red, or green colouring-matters. Nitrourea behaves in the same manner. The detailed examination only of one of these colours can throw any light on this reaction.

Boiled Milk.—Experiments undertaken by Dr. Chamouin, first on kittens and afterwards on infants, show that those fed on boiled milk thrive much better than those fed only on milk in its natural state. Not only does the boiling sterilise the milk, but it also renders it much more easy of digestion. The milk must of course be kept covered after it is boiled.—*The Public Health Journal* (New York), August, 1897.

A Rapid and Practical Method for Determining Carbon in Iron.—J. George Heid.—The sample is treated with copper ammonium chloride in the usual manner, and the separated carbon is collected on an asbestos filter, where it is washed successively with water, alcohol, and ether; it is then transferred to a Rose crucible, dried at 120° , and weighed. A stream of oxygen is led into the crucible which is heated over a Bunsen flame, and the carbon is thus burned off in from three to five minutes; the difference in the weight is the "total carbon." The "graphitic carbon" is obtained by dissolving the iron in dilute hydrochloric acid and determining the separated carbon as before.—*Eng. Min. Journ.*, lixiii., 64.

MISCELLANEOUS.

Improvements in the Colorimetric Tests for Copper.—George L. Heath.—Standard ammoniacal copper solutions which are permanent for long periods, may be prepared by replacing nitric acid by sulphuric acid after the solution of the pure copper in the former, provided a considerable excess of ammonia is added and the solution preserved in bottles with stoppers sufficiently tight to prevent any escape of ammonia. In the analysis of lean material for copper, a double precipitation of the iron and alumina by ammonia is found to yield better results, and in less time than either the precipitation by aluminium or a single precipitation by ammonia.—*J. Am. Chem. Soc.*, xix., 24.

International Conference of Leather-Traders' Chemists.—A Conference, as above, holden on Tuesday and Wednesday, Sept. 28th and 29th, at Herold's Institute, Bermondsey (Leathersellers Company's Tanning School), and at which Great Britain, the United States of America, Austria, Denmark, France, Germany, Norway, and Sweden were the countries represented, concluded its proceedings on the 30th ult., by a joint meeting of the leather trade and its allies at Leathersellers' Hall, kindly lent by the Worshipful Company of Leathersellers. The object of the Conference was to arrive at uniformity in the matter of tanning analyses, and formally establish an International Association of Leather-Traders' Chemists, &c. The Conference was opened by Mr. C. T. Millis, Principal of Herold's Institute, and representing the Governors of the Borough Polytechnic, of which the Institute is a Branch; the chair being afterwards taken by Dr. Perkin, F.R.S. The Rt. Hon. W. L. Jackson presided at the Leathersellers' Hall Meeting. As the first President of the International Association the Conference elected Mr. Alfred Seymour-Jones, F.C.S., F.I.C.; Hon. Secretaries, Prof. H. R. Procter (Yorkshire College, Leeds), and Dr. J. Gordon Parker (Herold's Institute, London).

The Cause of Arsenical Poisoning by Wall Papers.—Thomas Bolley, F.C.S.—It has long been recognised that arsenical wall papers do serious mischief, but the work of Gosio and of Emmerling seems to have cleared up that mystery which has surrounded the matter. Certain moulds, including the very common *muco* *mucosa*, have a remarkable property of decomposing arsenical compounds with the evolution of volatile products containing arsenic, and the highly poisonous character of volatile arsenical compounds, coming into the system by way of the respiratory organs, is well known. Arsenious acid is, even in small quantities, a highly antiseptic substance, and poisonous to moulds, so the throwing off of the arsenic in a volatile form may be an effort of nature to cast out the poison. The arsenical copper greens, and other colouring-matters containing arsenic, are still used, and, paradoxical as it may appear, it is by no means improbable that the most dangerous wall papers are those containing a mere trace of arsenic, as when the quantity is large the moulds cannot exist. Traces of arsenic may come into wall papers from the imperfect washing of vessels used to contain the more highly arsenical colours. Now that boric acid is very cheap, the old and perhaps forgotten suggestion of Bolley to use a precipitated borate of copper as a green pigment in place of the arsenical green deserves attention. Bolley's green is prepared by taking two parts of blue vitriol (crystallised cupric sulphate) and three parts of borax in separate quantities of cold water and mixing; after which the precipitate is washed and dried. Dyed and printed fabrics now very frequently contain traces of arsenic.—*Journal of the Society of Arts*.

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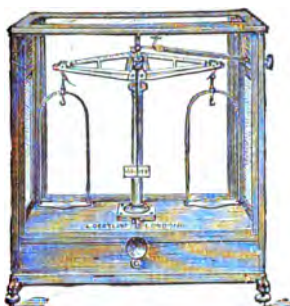
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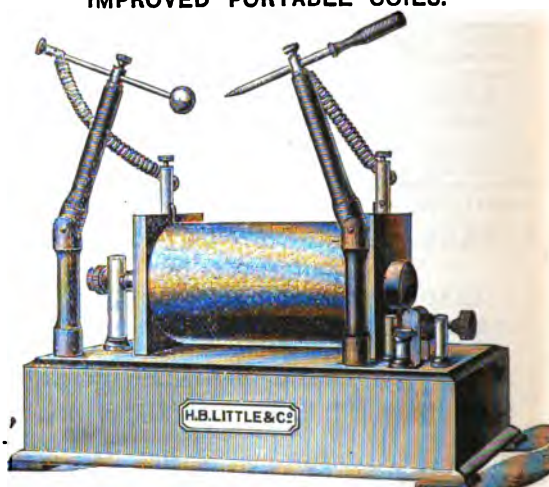
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THE CHEMICAL NEWS.

VOL. LXXVI. No. 1974. 5 1927

ON THE COMPOSITION OF CERTAIN CANADIAN VIRGIN SOILS.*

By FRANK T. SHUTT, M.A. F.I.C., F.C.S.,
Chemist, Dominion Experimental Farms.

Of the many investigations carried on by the Chemical Division of the Dominion Experimental Farms during the past ten years, not the least in scientific interest nor in agricultural value have been those which have had for their object the determination of the amounts of plant food in certain typical and virgin soils of the Dominion. The data are not as yet voluminous, for this work is one that consumes much time, and other and more pressing demands have only permitted an intermittent attention to it; nevertheless we have been able to place on record results which go far towards indicating the character of many soils representative of large untilled, or, at all events, but partially settled districts in Canada.

In all, we have submitted to complete analysis about ninety samples. These comprise surface and sub-soils taken from the Atlantic to the Pacific in the various provinces of the Dominion, and, to the best of our knowledge, from areas which had never been manured or cropped.

It is not my purpose to present in this paper all the data obtained, nor to attempt an interpretation of all the figures, chemical and physical, that have resulted from this work, for such would scarcely be possible.† My intention rather is to bring before you the percentage composition of these soils as regards certain of the more important elements of fertility, and to draw such deductions as to relative richness or deficiency in plant food as may seem warranted when comparing the figures with those obtained from the examination of soils in other countries.

The Value of an Ordinary Soil Analysis.

The exact value of a chemical analysis towards ascertaining the fertility of a soil is a question that probably will always be open to discussion, and all present are doubtless aware that no problem in agricultural science has excited more interest or been debated with greater warmth. We are obliged to confess that a knowledge of the amounts of nitrogen, potash, phosphoric acid, &c., as estimated by our present methods of determining "total" or maximum amounts of plant food constituents by strong solvents, is not in itself sufficient for making a diagnosis as to the crop-producing power of a soil. Why this is so will be apparent upon reflection. In the first place, hydrochloric acid of the strength employed in the analysis dissolves from the soil the mineral constituents in much larger amounts than are present in an immediately available condition; and, secondly, there are factors other than the amount of plant food present that are equally important in determining a soil's fertility. The physical condition of the soil,—including retentivity of moisture, capillarity, permeability, &c.,—the climatic conditions, including rainfall, mean temperature, sunshine, &c., must all be carefully considered in conjunction with the analytical figures when endeavouring to interpret the latter with a view of ascertaining a soil's probable crop-producing ability. The case is very similar to that of

water analysis, in which it is universally held that all possible information respecting the source and its environment must be in the possession of the chemist before he can intelligibly and correctly give judgment from his figures upon the quality of the water under examination.

It is often urged that our usual method of soil analysis, using hot, strong, hydrochloric acid as a solvent, only indicates the amounts of plant food that may become available, not the amounts that are immediately assimilable. This is true, and it is certainly a serious drawback, but it in no wise makes the results of no value, as some would have us believe. It gives, we may suppose, the maximum amounts of the mineral elements present which under the influence of favourable climatic and mechanical conditions may become useful to crops. It shows decisively deficiencies in any of the plant food constituents, if such exist, and consequently affords valuable information regarding the suitability of the soil for various farm crops, and further indicates the direction in which fertilisation may be economically and profitably carried on. Soils with large stores of plant food, even if such be partially or largely in a locked-up condition, have repeatedly been shown to have a greater agricultural value than those that furnish to the same solvent much less amounts. The probabilities are that, other things being equal, soils of the former class will contain, or at all events under favourable circumstances yield, larger amounts of readily assimilable food than those possessing smaller "totals" or maximums. Soils showing percentages of maximums above the average invariably prove fertile, if climatic influences are favourable. We cannot argue very closely, I admit, but from such an analysis we are able to predict possibilities as to productiveness, provided agencies favourable to the unlocking of soil plant food are present.

Soil Tests for ascertaining Available Plant Food.

Pot or plot experiments are as yet, we confess, the only tests that can infallibly indicate a deficiency in available fertilising constituents. Such methods consume much time, are cumbersome, and from their very nature scarcely suited to wide application. What is needed is a laboratory method or methods, in addition to those we now use, which will furnish data in accordance with the results obtained by actual soil trials with crops. This is a question that at present many agricultural chemists are engaged upon, and I venture to hope that ere long the renewed interest in this work will result in satisfactory methods being established, both for available mineral constituents and nitrogen.

Dr. Dyer's Work.

In March, 1894, Dr. Bernard Dyer's work on available plant food in soils appeared. It was the beginning of a new era in soil analysis. Since that date increased attention has been paid to this branch of research, and especially so on this continent. Every year sees new and interesting data, the results of the labours of agricultural chemists of the Experiment Stations of the United States. Dr. Dyer, it will be remembered, showed, among other valuable results, that the root sap and the exudation of rootlets possessed an acidity approximately equivalent to that of a 1 per cent solution of nitric acid. From this he argued that such a solution would have a solvent action on the mineral constituents of the soil similar and equal to that exerted by growing crops. Further, he showed that results obtained by this method were strictly in line with the deductions made from the data of actual field trials. He therefore proposed that this solvent should be used to determine available potash and phosphoric acid in soils. Workers in the United States, members of the Association of Agricultural Chemists, besides using this solvent, have proposed and worked with, during the past few years, other solutions, such as ammonium chloride and calcium chloride. None of these, however, have had the support or corroboration of experiments to show that they were similar or comparable in their action upon the soil to the

* Read before the British Association (Section B), Toronto Meeting, 1897.

† The data here referred to are to be found in the Reports of the Chemical Division of the Experimental Farms, 1888—1896.

solvent action of root exudations. Consequently they do not as yet appeal to agricultural chemists with the same force as the solvent proposed by Dr. Dyer.

Solvents employed.

The solvent used by us in the determination of "total" or maximum percentages of the mineral constituents has been hydrochloric acid, sp. gr. 1.115 (corresponding to 22.86 per cent HCl), 10 grms. of the air-dried soil being digested with 100 c.c. of the acid at the temperature of the water-bath for ten hours.

For the estimation of the "available" potash and phosphoric acid, 1 per cent citric acid solution has been employed, digesting 100 grms. of air-dried soil with 500 c.c. of the solvent for five hours at room temperatures.

Standards of Fertility.

It has been remarked that climate and the physical condition of a soil are potent factors in determining fertility. To this might be added the statement that fertility (i.e., crop-producing power) is a relative quality, depending to a large extent on the crop grown. The ability of plants to forage for and appropriate their food varies greatly, so that what might be an adequate supply of food for one might prove an insufficiency for another. Buckwheat and wheat will very well illustrate this variation in foraging and assimilating ability. For these reasons chiefly—for of course there are others—it is impossible to establish rigid standards as regards the minimum amounts of plant food for all crops that must be present in order that a soil may be classed as economically productive.

It is not impossible, however, using a large number of analyses of soils, the productive power of which soils is approximately known, to deduce percentages or limits in plant food, below which, under ordinary circumstances, soils may be considered as deficient or lacking, and above which they may be considered as well supplied or rich in the essential mineral elements. Professor Hilgard, of the California Experiment Station, the highest authority on American soils, considers that less than 0.09 per cent potash indicates a deficiency in this element, and that the limits of this constituent in good soils range approximately from 0.8 to 0.5 per cent in heavy clays, from 0.45 to 0.30 per cent in medium loams, and from 0.3 to 0.1 per cent in sandy loams. Regarding phosphoric acid, he says that 0.2 per cent is sufficient when associated with a good supply of lime, though it may in certain soils reach or exceed 0.3 per cent. Respecting lime, Hilgard states 0.1 per cent in sandy loams as the lowest limit for good crops, 0.25 per cent in clay loams, and 0.3 per cent in heavy clay soils.

Standards of Fertility in Canadian Virgin Soils.

Our data indicate that good agricultural soils in Canada possess usually between 0.25 and 0.5 per cent potash; less than 0.15 per cent, in our experience, points to the necessity, or at all events to the value, of potassic fertilisers, though with good climatic and soil conditions the limit might be reduced to that suggested by Hilgard.

The phosphoric acid in Canadian virgin soils of average fertility lies usually between 0.15 and 0.25 per cent. Some good soils contain from 0.25 to 0.3 per cent, and a few exceed the latter figure. The adequacy, or otherwise, of phosphoric acid in a soil would appear to depend largely on the accompanying amount of lime. Increased crop production has usually followed the application of phosphatic fertilisers to soils containing less than 0.15 per cent phosphoric acid.

Lime ranks next in importance to potash and phosphoric acid in a consideration of the mineral constituents of plant food. I am led to the conclusion that clay soils containing less than 0.1 per cent will have their productiveness increased by a dressing of lime in one or other of its agricultural forms. Peaty soils, and soils generally that are exceedingly rich in organic matter, are

frequently poor in this element. All such have been found to respond to an application of lime, and more particularly so when given in conjunction with potash and phosphoric acid. For these classes of soils, therefore, I deem it advantageous that they should contain at least 1 per cent of lime.

Richness in nitrogen may be measured to a large degree by the organic matter or humus content, though the condition or stage of decomposition of this organic matter is an important factor in determining nitrogen's availability. A large number of our good soils contain between 0.1 and 0.2 per cent, though many reach 5 per cent, and some exceed 1 per cent nitrogen.

In the following brief review of Canadian virgin soils I have not given any detailed data of their physical condition or composition, for the determinations in our laboratory have been confined simply to the separation of the mineral components into (a) clay and fine sand, and (b) coarse sand, according to the method of Schlessing. The results of this separation, together with remarks on the physical condition or tilth of the soils, have been indicated in general terms in discussing the samples. If it had been possible to have made a more extended physical examination I believe the data would have proved most valuable, for the degree of permeability of water and air, the relative size of the soil particles, compactness, water-holding capacity, &c., are important factors towards establishing a soil's suitability for the various agricultural crops.

(To be continued).

ANALYSIS OF SOME PRE-CARBONIFEROUS COALS.*

By W. HODGSON ELLIS.

THE occurrence of anthracite in the calciferous sand-rock of New York, near the base of the Lower Silurian, was recorded by Vanuxem in 1842 ("Geology of New York," iii., 33). It was found in cavities in the rock associated with quartz crystals, sometimes existing within the crystals. He stated that it contained "86½ per cent of carbon; 2 of light cream-coloured ashes, which were of siliceous; and 11½ per cent of water," i.e., volatile matter. He attributed its origin to the action of heat on bituminous matter contained in the rock.

Sterry Hunt subsequently described ("Geology of Canada," 1863, p. 524) a substance of like nature filling veins and fissures in rocks of similar age near Quebec and on the north shore of Lake Superior.

E. J. Chapman (*Canadian Journal*, vol. x., p. 410) described an anthracite from Lake Superior, occurring in a banded vein with quartz and iron-pyrites, an analysis of which gave—

Moisture	2.08
Volatile matter	3.56
Fixed carbon	94.36
Ash	0.00
	100.00

In his "Minerals and Geology of Central Canada," p. 143, Chapman proposed the name "anthraxolite" for this pre-carboniferous anthracite, chiefly because its mode of occurrence differs from that of the anthracite of the coal measures.

In *Bulletin No. 2* of the Ontario Bureau of Mines, November, 1896, the discovery is reported of a remarkable deposit of coaly material occurring as an irregular vein, about 9 feet thick, in black fissile seats of Cambrian age, situated near Sudbury, the locality of the famous

* Read before the British Association (Section B), Toronto Meeting, 1897.

Canadian nickel mines. The discovery roused great public interest, and hopes were entertained that the coal would prove of commercial value. Specimens were sent to Dr. Dawson and Mr. Hoffmann, of the Geological Survey of Canada, who reported it as consisting of anthraxolite and quartz, the latter constituting 55.95 per cent of the whole. Dr. A. P. Coleman was sent by the Ontario Government to examine the deposit, and he described it as a lustrous black mineral, forming "small plates or irregular cubical blocks, the largest observed being three-quarters of an inch square. Between the plates or cubes there is generally more or less quartz, and in some weathered portions on the surface the quartz remains as a porous cellular mass." Dr. Coleman considered that "the source of the anthraxolite is probably bituminous matter contained in the adjoining beds of slate. By metamorphic action most of the volatile matter has been removed from the once fluid or plastic bitumen, leaving the present cracked and quartz-cemented anthraxolite."

In the *Proceedings of the Canadian Institute* for February, 1897, will be found a detailed account of this interesting deposit by Mr. G. R. Mickle, and also a chemical analysis by Mr. W. Lawson and myself.

The pure coaly substance has a specific gravity of 1.865 and a hardness between 3 and 4. It burns with difficulty, giving off a good deal of heat (a sample containing 3.99 per cent ash gave, in Fischer's calorimeter, 7,490 calories per gram.) and leaving a light fawn-coloured ash consisting of silica with a trace of oxide of iron. The quantity of ash varies in different samples, some containing as high as 60 per cent. The average as far as yet examined is about 20 per cent.

The following is the proximate analysis of an average and of a selected sample:—

	Average.	Selected.
Moisture	4.0	4.0
Volatile matter	1.3	1.8
Fixed carbon	74.2	90.1
Ash	20.5	4.1
	100.0	100.0

The ultimate analysis of a carefully picked specimen, freed from moisture, was as follows:—

Carbon	94.92
Hydrogen	0.52
Nitrogen	1.04
Sulphur	0.31
Oxygen	1.69
Ash	1.52
	100.00

The striking point in this analysis is the small percentage of hydrogen, which is less than that given in any published analysis that I have been able to find of anthracite of the carboniferous age.

In this respect, as well as in its physical properties, it bears a striking resemblance to the mineral schungite described by Inostranzeff as occurring in Huronian rocks on the shore of Lake Onega, in Russia. This mineral has a specific gravity of 1.84 and a hardness of 3.5 to 4, and contains 99.2 per cent of carbon and 0.4 per cent of hydrogen. The Sudbury coal contains more oxygen, but otherwise the resemblance between the two is very striking. The so-called graphitoid of Sauer, from the Saxon Erzgebirge, with 99.8 per cent of carbon and 0.2 per cent of hydrogen, is another closely allied mineral.

It seemed of interest to compare the composition of the Sudbury anthraxolite with that of specimens from other Canadian localities, and accordingly Mr. Lawson and myself made an analysis of a specimen from the neighbourhood of Kingston, for which we were indebted to Mr. W. G. Miller. It occurs in a vein of barite cutting Lower Silurian limestones. The anthraxolite coats and fills

crevices in the barite. The mineral is duller and softer than the Sudbury anthraxolite. Its specific gravity is 1.365. The analysis of the dry substance gave the following figures:—

Carbon	90.25
Hydrogen	4.16
Nitrogen	0.52
Sulphur	0.66
Oxygen	3.69
Ash	0.72
	100.00

Since then, through the kindness of Dr. G. M. Dawson, Director of the Geological Survey of Canada, I have had the opportunity of examining three other specimens of anthraxolite—one from Cap Rouge, near Quebec, and two from the peninsula of Labrador.

Anthraxolite from Cap Rouge.—A black coaly substance occurring in cracks of "beds of a hard jaspery character," associated with black bituminous shales of Lower Silurian age ("Geology of Canada," 1863, p. 203).

The analysis of this specimen was as follows:—

Moisture	0.19
Ash	9.02
Carbon	82.90
Hydrogen	5.50
Oxygen	2.37
	100.00

(Including nitrogen and sulphur not determined).

Anthraxolite from Lake Mistassini.—A bituminous mineral, with the lustre and colour of anthracite, filling cavities in calcite veins in limestones of Cambrian age (A. P. Lowe, "Report of the Geological Survey of Canada," 1895, 267 L.). I found the composition of this specimen to be—

Moisture	1.75
Ash	1.07
Carbon	92.71
Hydrogen	1.02
Oxygen, &c.	3.45
	100.00

Anthraxolite from Lake Petitsikapau, District of Ungava, Labrador Peninsula.—In veins, with quartz in Cambrian shales. Foliated with plates at right-angles to the walls. Collected by Mr. A. P. Low. A purer specimen gave Mr. Hoffmann ("Report of the Geol. Survey of Canada," 1894, 66 R.) the following results on proximate analysis:—

Water	3.56
Volatile matter	2.48
Fixed carbon	86.83
Ash	7.13
	100.00

My specimen had the following composition:—

Moisture	0.81
Ash	48.37
Carbon	49.39
Hydrogen	0.67
Oxygen, &c.	0.76
	100.00

It will be seen that in none of these is the hydrogen so low as in the Sudbury anthraxolite. It will also be seen that they vary among themselves as much as do the varieties of coal which occur in the beds of the carboniferous period. This variability is in strict accordance

with the view that they result from the metamorphosis of bitumen, of the various stages of which metamorphosis they serve as excellent illustrations. To make this more striking, I have calculated the analysis to the dry ash free substance, and for the sake of comparison I have added the analyses of petroleum, asphalt, and albertite—the latter a coal-like mineral occurring in a vein in rocks of lower carboniferous age in Nova Scotia, for some time successfully worked as fuel. At the other end of the scale I have given the analyses of schungite and graphitoid.

	Locality.	Carbon.	Hydro- gen.	Oxygen, &c.
1. Petroleum ..	Pennsylvania ..	82.0	14.8	3.2
2. Asphalt ..	Mexico ..	83.3	10.8	5.9
3. Albertite ..	Nova Scotia ..	86.0	9.0	5.0
Anthraxolite .	Kingston ..	90.5	4.2	5.5
"	Cap Rouge. ..	91.3	6.2	2.5
"	Lake Mistassini	95.2	1.2	3.6
"	Sudbury ..	96.4	0.5	3.1
"	Lake Petitsikapau	97.1	1.3	1.6
4. Schungite ..	Lake Onega ..	99.2	0.4	0.4
5. Graphitoid ..	Erzgebirge. ..	99.8	0.2	0.0

1. Ste.-Claire Deville, *Comptes Rendus*, lxi., 442.
2. H. Endemann, *Journ. Soc. Chim. Ind.*, xv., 872.
3. Wetherill, *Trans. Am. Phil. Soc. Philad.*, 353, 1852.
4. Inostranzeff, "Neues Jahrbuch für Mineralogie," i., 97, 1880; i., 92, 1886.
5. Sauer, *Zeit. Geol. Gesell.*, xxxvii., 441, 1885.

From these latter to graphite, the transition is rather one of crystalline form than of chemical composition, as indicated by analysis.

Chemical Laboratory,
School of Practical Science, Toronto,
August 18, 1897.

THE CARBOHYDRATES OF THE CEREAL STRAWS.*

THE work upon the barley crop of 1896, which was reported in outline to the Chemical Section in a paper read by Mr. Cross, has been more fully dealt with in a paper read subsequently, and published in the *Journal of the Chemical Society*, 1896, pp. 804—818. The subject was also dealt with from the more special point of view of the relation of the furfuroic constituents of these straws to the important problems of animal digestion and alcoholic fermentation in a paper published in the *Journal of the Fed. Inst. of Brewing*, 1897, Pt. 1.

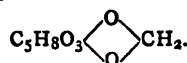
The investigations have been continued without interruption. We have further and more closely studied the products of acid hydrolysis of the cereal straws and of the celluloses isolated from them, and the main results of these researches are embodied in a paper read at the meeting of the Chemical Society, London, on June 17.

Generally the results of the preceding paper (*loc. cit.*) are amplified and confirmed. As it had been previously shown that the furfural-yielding constituents of fodder plants are in large measure hydrolysed and assimilated by the animal organism, so the evidence is accumulating that certain of these compounds when fully hydrolysed (to monoses) by artificial processes are susceptible of alcoholic fermentation.

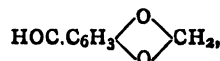
It having been finally established that the pentoses themselves are entirely resistant to the attack of the yeast cell, it follows that we are dealing with a class of furfural-yielding carbohydrates, *not* pentoses.

At the present time the reactions of these compounds

clearly indicates that they are pentose-derivatives, and most probably methylene ethers of the C₅ sugars of the general formula—



It is difficult to devise reactions of decomposition or synthesis by which such a constitutional formula could be finally verified. The literature of the analogous compound diperonal—



but with an aromatic in place of a pentose residue, may be cited in evidence of the exceptional difficulty of the problem presented.

The authors are glad to report that through the kindness of friends they have now access to a vessel enabling them to operate upon a large weight (7 kilos.) of the raw materials.

Working upon this extended scale, and upon the basis of the results established by long investigation and previously reported to the Section, we may confidently expect more positive and, we hope, final results.

ANALYSIS OF A BLACK SILK DRESS.

By Dr. T. L. PHIPSON,
formerly of the University of Brussels and the Laboratoire de Chimie
Pratique, Paris.

A LADY paid a visit to my laboratory a short time ago and enquired whether I had ever made an analysis of a silk dress. I confessed that among the many hundreds of various things examined in the course of a long series of years, I had only been called upon hitherto to discover the nature of certain poisonous colours applied to silk gloves and silk stockings, but I could find no analysis of a silk dress on my books.

Nevertheless, my fair visitor was anxious that I would undertake this analysis for her, and also expressed the desire that I would publish the results in the *CHEMICAL NEWS*, a journal which, she said, she was in the habit of reading.

Her husband was interested in the silk trade, but had little knowledge of chemistry, whilst she herself had gone through a course of practical science, and wished to convince him that the value of silk material might be ascertained by analysis. She explained, moreover, that the material she placed in my hands was intended for a silk blouse, that it was a medium quality of silk (neither the most expensive nor the cheapest), and she had heard that certain black silks, when stored in large bulk, in dry hot weather, were liable to spontaneous combustion, that two such cases were already known (one in Paris and one in New York), and she was therefore anxious that her husband should increase his insurance upon his stock of silk goods.

The following days I devoted myself to the work in question, and I found that the material contained a very large amount of substance that was not silk at all; in fact, that it was considerably "weighted," as all silks are to a greater or less extent. It would not burn with flame, but smouldered away like tinder and left a large amount of ash, the principal ingredient of which was oxide of tin. Indeed, I have examined specimens of poor tin ore from Cornwall that did not contain more tin than this material for a lady's blouse; and I at once realised the fact that the silk dresses worn by the ladies we see daily parading in Regent Street and Bond Street, taken together, would represent a Cornish mine of very fair quality.

But this enquiry has brought to light a new and powerful application of chemical analysis—already one of

* Report of the Committee, consisting of Professor R. Warington (Chairman), C. F. Cross (Secretary), and Manning Prentice. (Drawn up by the Secretary). Read before the British Association (Section B), Toronto Meeting, 1897.

the greatest powers in the hands of man—namely, that the duration or wear of a silk dress can be determined by analysis. It used to be said that a garment of pure silk would last a life-time; but I happen to be acquainted with a very clever young milliner who has assured me that the silk material I have examined, if worn every day, would not last more than three months—meaning, of course, that by that time it would be “utterly shabby, greasy-looking, and showing the threads.”

The figures obtained in my analysis are as follow:—

Water	11.43
Ash (mostly tin oxide and silica)	14.30
Real silk	28.14
Organic matters, &c., not silk	46.13
	100.00
Nitrogen	4.76

The weighting of silk is now carried on to so great an extent in France, Germany, and Switzerland, that some foreign silks get shabby with a few weeks' wear; and we are seriously told that the public prefer these cheap products, as the fashions of jackets, blouses, and skirts change so rapidly that it would be useless to purchase silk of better quality!

Casa Mia, Putney, S.W.

A MODIFICATION OF THE CYANIDE TITRATIONS OF COPPER.

By HARRY BREARLEY.

This paper is offered with some diffidence; its subject-matter has been so often examined and discussed by those daily interested in its accuracy. A belief that the modification is also an improvement, and its general bearing on a subsequent paper must stand as an apology for any seeming presumption on the part of one only rarely engaged with copper estimations.

For each element in the “alkaline acetate” separations the question—“How shall the separated element be estimated?” had in all cases to be primarily settled. For obvious reasons, when a large number of estimations had to be made a volumetric process was preferred.

The cyanide method of Parkes and the iodide method of De Haen are, of all volumetric methods for copper, perhaps the most widely known and practised. Both, however, are at a disadvantage when the copper percentage is low and the bulk of the solution also large. This being so, the proposed acetate separations of iron and copper would entail an additional operation before the estimation could be made, and thus destroy for the particular purpose a volumetric method's chief charm—rapidity.

The latter stages of the cyanide titration are rather indistinct. The solution assumes a pale violet or lavender tint, which fades very gradually independently of any cyanide additions; and so it becomes not altogether impossible, in following the changes—unconsciously, of course—to have one's expectations warping one's judgment. The suggestion to titrate only to a pale lavender tint (Clowes and Coleman, “Quant. Anal.,” 2nd edition, p. 195) does not greatly amend this uncertainty.

The proposed modification lies in the application of the silver iodide indicator to the usual titration.

If the silver iodide be formed in the solution before the cyanide is added it is no indicator whatever; because the suspended iodide is equally or more active to the cyanide than is the ammoniacal copper, and so the solution clears sometime before the blue colour is discharged. This difficulty is obviated by adding cyanide to the usual point, or thereabouts, and then adding potassium iodide

and going back to a permanent turbidity with silver nitrate. As a matter of fact, the turbidity is not really permanent, but only persistent; because, on standing, any turbidity would disappear on account of its greater affinity for the cyanide, and a corresponding amount of copper being displaced would reproduce the characteristic blue colouration.* By this means, the end-reaction becomes certain, and with identical liquids gives good results. In the presence of additional compounds it exhibits its usual waywardness.

In 1888, Mr. J. L. Davies (CHEMICAL NEWS, lviii., 131) very modestly suggests the substitution of soda carbonate for ammonia. The carbonate is to be added in such excess as to partly dissolve the precipitated copper, and then the usual end-reaction is relied on. Mr. Merry modifies the original idea by adding enough tartaric acid to re-dissolve the precipitated carbonate to a clear blue solution, and then titrating. Eighteen months later (CHEMICAL NEWS, lxi., 183), Mr. Fessenden suggests a like change, especially stipulating that the soda carbonate be added to a solution containing free nitric acid, so as to completely effect the re-solution of the precipitated copper.

It was claimed for this modification that the final colouration did not lag, as with ammonia, and that the values are not so greatly affected by additional alkali or alkaline salts.

These suggestions occupy only two short letters to the editor; and, judging from the persistent way in which the ammonium titration is still practised and recommended, seem to have been undeservedly neglected.

A mere comparison of similar solutions made alkaline with ammonia and soda carbonate respectively, shows the latter under unfavourable conditions. But an addition of cyanide to the latter both changes and deepens the tint, and this deepening keeps pace with the progressing titration for some time. It cannot even be pretended that the colour vanishes like phenolphthalein in alkalimetry, but it is no exaggeration to say that its sensitiveness is two or three times greater than when ammonia is used.

There are arranged below some titrations with soda carbonate and with ammonia under varying circumstances. Attention is chiefly paid to the former on account of its evident superiority.

The iodide indicator cannot be added before the titration with the soda any more than with the ammonia solution, although the turbidity holds out much longer in the former case; and the copper having once combined with the cyanide is not decomposed by the silver iodide turbidity.

It should be noticed that if cyanide be added to an alkaline solution of copper until the colour is just discharged, the whole of the cyanide has not combined with the copper. For instance, a solution containing 0.03 gm. of copper, if titrated as usual, would contain as “surplus” cyanide about 10 per cent of the total quantity used. Of course, by prolonged titration this surplus could be materially decreased, but—within reasonable time—it cannot be eliminated altogether.

For the following tests the cyanide is added until the colour is discharged, or very nearly so, and the solution allowed to stand five or ten minutes. Prolonged standing very slightly increases the amount of cyanide used, or more intelligibly perhaps, decreases the surplus. The bulk of solution throughout is 250 c.c., the amount of copper 0.02 or 0.03 gm.

* The following remarks by Field (CHEMICAL NEWS, i., 26) are interesting:—“When a soluble cyanide is added to a solution of a salt of copper, in a considerable excess of ammonia, until the liquid is perfectly colourless, the addition of nitrate of silver restores the deep blue colour, and an argento-cyanide of ammonia or of ammonia and copper is formed. There are a great variety of compounds consisting of silver, copper, cyanogen, and ammonium; one especially crystallises in splendid dark blue octahedra, and contains more than 60 per cent of silver. Another compound separates in pearly scales and contains a larger proportion of copper.”

Influence of Alkali.

Excess of ammonia ..	5	10	15	20 c.c.
Nett KCN	27.7	27.35	26.55	26.12 "
Excess of soda carbonate ..		10	20	40 "
Nett KCN		20.42	20.65	20.75 "

The strength of the alkalis were—Ammonia, 0.880, diluted to four times its bulk; soda carbonate, 200 grms. per litre. The results in the ammonia series seem to be at variance with common experience; they are not really so. The amount of cyanide needed to discharge the colour did increase with the volume of the ammonia, but in a greater degree the needful surplus cyanide also increased, and hence the reversed order of progression.

With an excess of only 5 c.c. of dilute ammonia, a final turbidity equivalent to one or two tenths of a m.grm. of copper will persist for several hours. As the ammonia increases in amount this time would decrease until, with an excess of 40 or 50 c.c., it becomes impossible owing to the rapid interchange of silver and copper, to observe the disappearance of the last traces of free cyanide other than by the returning blue colouration.

Influence of Volume.

Volume titrated ..	100	200	300 c.c.
Soda	21.16*	20.42	20.01 " KCN nett.
Ammonia	19.13	18.05	17.37 " " "

When the sample marked with an asterisk was made up to about 300 c.c. with water, the final turbidity disappeared, and to reproduce it required silver nitrate equivalent to 1.02 c.c. KCN, which subtracted from 21.16 brings the value into line with the "300 c.c." sample. The corresponding ammonia sample behaved similarly, but not with so great accuracy.

Proportion of Copper.—The volume of the solution was constant, 200 c.c.

Copper	0.005	0.01	0.02	0.03 grm.
Soda	4.85	10.1	20.4	30.67 c.c. KCN
Ammonia	—	8.8	18.05	27.5 " "

Excess of Cyanide.—These tests are intended to show the influence of adding more or less cyanide than a proper amount. This irregularity might happen either inadvertently or otherwise.

	I.	II.	III.	IV.
Total cyanide added	25.5	30.5	37.0	40 c.c.
Nett cyanide	21.6	25.95	28.55	28.95 "

This is an ammonia series. Test I. was decidedly, and test II. very faintly, coloured on adding the silver nitrate. The very considerable variation in the nett cyanide make the proposed iodide indicator of lessened value; because any error in noting the colour indication—the uncertainty of which provides the only excuse for using the AgI—would be only partially corrected by the added silver salt. The soda titration under like conditions is almost free from this error. Thus—

Total KCN	25.1	27.0	30 c.c.
Nett	20.5	20.6	20.78 "

This degree of constancy, taken in conjunction with the increased delicacy of the decolourisation, makes the end-reaction of a soda titration as delicate as the silver iodide is known to be under the most favourable circumstances. It is indifferent also to the soda titration in what manner the cyanide is added. Thus, when the required amount was run in quickly and then shaken, the net result was 20.38 c.c. KCN; when added in drops, shaking meanwhile, the result was 20.33 c.c. KCN

Alkali Chloride.

Ordinary HCl as Na(Am)Cl. C.c.

	0	5	10	20
Soda	20.4	20.4	20.52	20.52 c.c. KCN
Ammonia	28.41	31.4	32.65	33.3 " "

Alkali Nitrate.

HNO₃ (1.20) as Na(Am)NO₃. C.c.

	0	5	10	20
Soda	20.4	20.38	20.43	20.61 c.c. KCN
Ammonia	18.05	19.62	20.32	— " "

With the larger amounts of soda chloride and nitrate the final turbidity is, in the former case very slightly, and in the latter unmistakably, ill-defined. Only the nitrate deserves more than a passing notice.

The whole of the variations at present under consideration were made on a solution containing an exaggerated indicator, but no copper. They were all without appreciable influence, except soda nitrate. The nature of the difficulty is set forth in the table.

HNO₃ (1.20) as NaNO₃. C.c.

		5	10	20
Excess soda carbonate {	5 c.c.	6.3	7.27	7.5
	10 "	—	6.37	7.12
	20 "	—	6.35	6.35

The values are c.c. KCN. The table also points to the remedy, namely, an increasing excess of soda carbonate with increasing quantities of soda nitrate. Any error due to this irregularity could be detected by adding more soda carbonate. In case there was a previous insufficiency, the turbidity would clear, and then titration would finish in the usual way. A proper titration is not appreciably affected by the further addition of carbonate.

Alkali Acetate.

33 per cent acetic acid as acetate. C.c.

	0	5	10
Ammonia	29.1	31.2	32.1 c.c. KCN

Grms. crystal. soda acetate.

	0	2½	5	10
Soda	20.35	20.4	20.63	20.63

The most casual comparison of the two series of results greatly favours the soda cyanide process. R. T. Thomson, in a paper on "The Interference of certain Metals with the Accuracy of the Cyanide Estimation of Copper" (CHEMICAL NEWS, xxxiii., 152), shows that in ammoniacal solutions the salts of sodium and potassium have no appreciable effect. J. J. and C. Beringer (CHEMICAL NEWS, xlviii., 111, reprint from the *Proceedings of the Cornwall and Devon Miners' Association*) come nearer still to discovering the advantage of complete soda titrations. They say:—"It seemed to us that the interference of the ammonia salts is due to a reaction with potassic cyanide by which ammonium cyanide is formed, and that the excess of cyanide is required either because ammonium cyanide decomposes more rapidly or because it is less active than the corresponding potassic salt. In this case, the use of soda or potash for neutralising the excess of acid used for dissolving the ore would eliminate the disturbing element." And subsequently they show that the sulphates, nitrates, and chlorides of potash and soda have little or no effect on the cyanide titration; but the advisability of avoiding ammonia altogether does not seem to have suggested itself to them.

It has been previously noticed that Mr. Davies performs the titration without completely re-dissolving the precipitated carbonate, Mr. Merry effects the complete solution with tartaric, and Mr. Fessenden with nitric acid. So far as the colour reaction is concerned, it may be perfectly indifferent which acid is used. But if the iodide end-reaction be adopted, it is no longer an indifferent matter. One objectionable feature of nitrates has been already pointed out. There is another; and, if nitric acid is used alone, a more unpleasant one.

If four vessels containing solutions of copper acidified respectively with nitric, acetic, sulphuric, and hydro-

chloric acid are treated with an excess of soda carbonate, each will hold a clear solution. Let the solution be titrated with cyanide, and then, on the clear and colourless solution, after adding potassic iodide, carefully superstratify* dilute silver nitrate. In the first solution (nitric) the silver iodide will lose its lemon colour and become a dirty brown. The same change will take place decreasingly in the acetate and sulphate solutions. When these turbidities are dissolved by shaking they produce a more or less inky tinted solution, and make the final turbidity less easily recognisable. Hydrochloric acid is perfectly free from this defect.

In case the salts producing—or, perhaps better, allowing—this dirty colouration to be produced (because it is not noticeable except in presence of copper), are unavoidably present, the addition of soda chloride will ensure a clean turbidity. The inky tint is not permanent, but it disappears very slowly.

Interference of certain Metals.

The interference of nickel, cobalt, zinc, mercury, silver, and—less noticeable because of rarer occurrence—gold, platinum, and palladium, is known to be of such extent as to invalidate the cyanide titration of copper. Indeed nickel, cobalt, mercury, and silver may be quantitatively estimated by these means. There are, however, a large number of compounds whose effect it would be well to study anew under conditions which are altogether independent of colour reaction. The presence of those compounds which form a precipitate either before or during the titration are especially noteworthy in this connection.

Only the following three metals have been already experimented with. In each case 0.05 grm. of copper was present.

Iron.—Added as Fe_2Cl_6 .

0	0.01	0.03	0.05 grm. Fe
50.3	50.4	50.35	50.0 c.c. KCN
0.0500	0.0501	0.0500	0.0496 grm. Cu
—	0.0001	0.0000	0.0004 Error.

It is quite impossible under these conditions to make any use of colour reaction. The precipitate remains suspended for some time, and is so finely divided as to pass through a moderately porous paper. An expansive asbestos filter gives a clear filtrate.

Aluminium.—Added as Al_2Cl_6 .

0	0.1	0.3	0.05 grm. Al
50.3	50.6	51.1	51.1 c.c. KCN
0.0500	0.0503	0.0508	0.0508 grm. Cu
—	0.0003	0.0008	0.0008 Error.

The colour reaction can be distinctly followed. The variations accord with ammon. cyan. titration when performed as usual.

Manganese.—Added as MnCl_2 .

0	0.01	0.03	0.05 grm. Mn
50.3	47.35	47.3	47.4 c.c. KCN
0.0500	0.0471	0.0470	0.0471 grm. copper
—	0.0029	0.0030	0.0029 Error.

The colour change is no guide. The results are in accord with those by the usual titration.

Considerable attention has been paid to the cause of these low values, though without, so far as I know, any satisfactory explanation being come to. The deficiency here is somewhat less than the usual amount. Thus Field (CHEMICAL NEWS, i., 63) finds a deficiency of 12 per cent, Thomson a deficiency of 20 per cent.

It is noteworthy that the deficiency seems to be inde-

pendent of the amount of manganese present. No especial emphasis is laid on this point. The tests were arranged merely to decide whether or no the manganese would act as usual, and, although interesting in the above character so far as they go, they are altogether inadequate to support a far-reaching pronouncement.

A more extended list of added metals will be given later, together with some means of obviating the interference and applying the corrected process to the determination of copper in copper alloys.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING AUGUST 31ST, 1897.

By SIR WILLIAM CROOKES, F.R.S.,

and

PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, *Metropolis Water Act*, 1871.

London, September 10th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 175 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from August 1st to August 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in a previous report.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 175 samples examined during the month all were found to be clear, bright, and well filtered.

The rainfall at Oxford during August was 3.94 inches, and was very fairly distributed, with the exception of 0.88 inch which fell on the 30th; the average fall for the last 30 years during this month is 2.32 inches; we have therefore had an excess of 1.62 inches. This figure is also the actual excess for this year, to the end of August.

Our bacteriological examination of 235 samples taken by us have given the following results; we have also examined 41 other samples, from special wells, stand-pipes, &c., making a total of 276 in all:—

	Microbes per c.c.
Thames water, unfiltered (mean of 25 samples)	3420
Thames water, from the clear water wells of five Thames-derived supplies (mean of 117 samples)	51
Ditto ditto highest	775
Ditto ditto lowest	10
New River, unfiltered (mean of 25 samples) ..	3420
New River, filtered (mean of 23 samples) ..	44
River Lea, unfiltered (mean of 25 samples) ..	5846
River Lea, from the clear water well of the East London Water Company (mean of 21 samples)	64

The fact that, in spite of the great excess of rain, the number of microbes per c.c. has decreased in the case of every Water Company's supply, except one, which remains the same as during the previous month, shows

* I have elsewhere pointed out that the rapidity with which the resulting turbidity disappears indicates the approaching end-reaction.

that the filtering appliances of the companies are in excellent working order.

We are, Sir,

Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

THE ACTION OF NITRIC ACID ON TRIPHENYLMETHANE.

By E. S. SMITH.

THE method of preparing trinitrotriphenylmethane by the action of fuming nitric acid on triphenylmethane is described by Fischer (*Ann. Chem., Liebigs*, cxciv., 254). In an attempt to make this preparation I departed from the exact directions of Fischer, and obtained a substance shown by analysis to be triphenylcarbinol, $C_6H_5(C_6H_4)_3C.OH$. Following are the details of the work:—I placed in a flask an unweighed quantity of triphenylmethane recently crystallised from benzene, and probably still retaining a little benzene; poured in an unmeasured quantity of nitric acid (sp. gr. 1.34), and then some fuming nitric acid; allowed the mixture to stand a short time, then heated on the sand-bath for a few minutes. After the reaction was ended a large amount of water was poured in. The reddish yellow precipitate formed was filtered off on a platinum cone. By pressing down the precipitate a small amount of a red oil, having the smell of nitrobenzene, was forced out. The solid substance was purified by treating it with hot glacial acetic acid, crystallising it from a mixture of benzene and glacial acetic acid, recrystallising from benzene, and finally from alcohol. The purified substance is white, resembles triphenylcarbinol, and melts sharply at 161° (uncorr.). A test for nitrogen by the Prussian-blue method showed absence of nitrogen.

0.138 grm. gave 0.0773 grm. water, and 0.4458 grm. carbon dioxide, indicating the following composition:—

	Calculated for triphenylcarbinol.	Found.
C ₁₉	87.80	88.10
H ₁₆	6.15	6.22
O	6.15	—
	100.00	

Several attempts to confirm this work by repeating it were, on the whole, unsuccessful, though various modifications in the strength of acid, manner of heating, &c., were tried. In one case a very small quantity of a white crystalline substance was obtained, which, after recrystallising from benzene, melted at 161° .

Nitric acid of sp. gr. 1.42 does not act upon triphenylmethane until the temperature is raised to about 100° ; but the product obtained in this case, as in most of the other experiments, was a waxy substance of salmon colour, from which I was not able to get a crystalline compound.

I could find no record of triphenylcarbinol having been made in this manner; the usual method of preparation is to oxidise the triphenylmethane with chromic acid.—*American Chemical Journal*, xix., 702.

Condy's Fluid.—Notice of Removal.—The Proprietors of "Condy's Fluid" notify that they find it necessary to remove to more extensive and commodious premises. All communications should in future be addressed to Condy's Fluid Works, 65, Goswell Road, London, E.C.

PROCEEDINGS OF SOCIETIES.

THE CHEMICAL AND METALLURGICAL SOCIETY OF SOUTH AFRICA.

Meeting held on July 17, 1897, at Johannesburg.

Mr. CHAS. BUTTERS, President, in the Chair.

THE PRESIDENT delivered his Inaugural Address, in which he first referred to the history of chemistry and metallurgy on the Johannesburg gold-fields. Until now there has been but one industry, viz., gold-mining. Properly speaking there is no Chemical Industry, as, whatever chemical processes there may be at work, all have to do intimately with the extraction of gold. Silver and copper are both at the Willows and the Albert silver-mines, and silver-lead at the Transvaal silver-mines; these mines, however, have not, owing to the excessive cost of production, been yet made to pay. An attempt is being made to establish the manufacture of sulphuric acid, but the only factory yet at work produces acid of low commercial strength only. This is an important matter, as it will largely influence the production of cheap and trustworthy dynamite. Glass is being manufactured successfully at Pretoria. The coal in the Transvaal may be considered as practically inexhaustible, but the percentage of ash is extremely high, viz., 20 to 25 per cent, and the quality is very variable. Galena is very plentiful in the Transvaal, going as high as 80 per cent of lead; the smelting of the ore is done locally, but the demand is limited. But, as was pointed out above, the one industry that can, to a certain extent, survive the repressive measures of the authorities, is gold-mining, and it is to gold only that the country can at present look for commercial prosperity. A great deal of money and thought is now being expended on recovering the last traces of the precious metal which only a few years ago was allowed to run to waste; tailings have been regularly treated, and now managers are turning more attention to slimes.

Mr. D. J. WILLIAMS's paper on "Some Notes of the Estimation of Lead in Slags" was next discussed, after which followed the discussion of Mr. E. H. JOHNSON's paper "On the Reduction of Zinc-Gold Slimes."

Mr. CALDECOTT mentioned that he had used acid sulphate of soda or potash instead of sulphuric acid for this operation; he found it cheaper and better.

Mr. MCBRIDE, after paying tribute to the excellence of Mr. Johnson's paper, could not agree with him as to the increased cost of the acid process; he found three-quarters of a pound of acid enough for one pound of moist zinc-gold chips. He then gave details of his own system of cleaning up, which is so intimately connected with the acid treatment as to be almost inseparable from it in a discussion of this kind.

Mr. CROSSE had noticed that some bars assayed as high as 825 fine, while others went very much lower; he therefore analysed some, and found iron, nickel, cobalt, and lead, the three latter not being removable by sulphuric acid.

Mr. J. R. WILLIAMS then read a paper "On the Treatment of Battery Slimes." It is four years since the author first commenced experimenting on the recovery of gold from battery slimes, and he has now succeeded in evolving a process which works in a most satisfactory manner. Briefly it is as follows:—After separating the tailings, the slime water flows through a launder, where enough lime (in the form of milk of lime) is added to precipitate the slimes in a flocculent form; regularity of feeding the lime plays an important part in this process; an excess is as bad as too little. After mixing, the liquid passes through a series of large settling pits, 10 feet deep, from whence the water runs away practically clear. The sediment, with about 10 per cent of water, is then

pumped up, and treated with a 0.61 per cent solution of cyanide, and the gold recovered in the usual manner. The profit made during the months of April and May on this process alone was equal to a 15 per cent per annum dividend on the capital of the Company (Crown Reef G. M. Co.). The discussion of the paper was postponed.

Mr. W. A. CALDECOTT then read the following paper: "*The Solution of Gold in Accumulated and other Slimes.*" When battery slimes are settled in dams or pits, certain reactions take place which result in the formation of decomposition products, among which is ferrous sulphide, FeS , derived from the decomposition of pyrites, FeS_2 . This compound imparts a dark grey or black colour to the slimes where it occurs in any quantity, and is easily identified by the smell of sulphuretted hydrogen given off on the addition of acid. The exact stages of the decomposition may be open to discussion, but the author inclines to the opinion that the main stages of increasing oxidation are as follows:—

1. FeS_2 Iron pyrites.
2. $\text{FeS} + \text{S}$ Ferrous sulphide and sulphur.
3. $\text{FeSO}_4 + \text{H}_2\text{SO}_4$.. Ferrous sulphate and sulphuric acid.
4. $\text{Fe}_2(\text{SO}_4)_3$ Ferric sulphate.
5. $2\text{Fe}_2\text{O}_3, \text{SO}_3$.. Insoluble, basic, ferric sulphate.
6. Fe_2O_3 Ferric oxide.

During the treatment of slimes by cyanide, the presence of finely divided ferrous sulphide causes abstraction of oxygen from the solutions, whereby the solution of gold is prevented. Other ferrous compounds, such as ferrous hydrate, react in the same manner, as does also the decomposing organic matter always present in slimes. The obvious way to prevent this interference is to supply oxygen, either by pumping in air and agitating, or by chemical means; the cheapest and best of the latter is found to be permanganate of potash, about one-eighth to half a pound per ton of dry slimes was found to be sufficient. Speaking generally, old slimes are considerably more acid than old tailings, and a consumption of from 8 lbs. to 20 lbs. of lime per ton of dry slimes is by no means uncommon. The use of air is far cheaper, and in some cases it is an absolute necessity, as the gold simply will not dissolve until after aeration, so that, instead of merely accelerating the action of the cyanide, it becomes as necessary as that substance itself. After a few remarks the discussion of the paper was postponed and the meeting adjourned.

NOTICES OF BOOKS.

Patents for Inventions. Abridgments of Specifications Class 1.—Acids, Alkalies, Oxides, and Salts. Inorganic Period, A.D. 1884—88. Published at the Patent Office 1896. Price 1s.

THE Patent Laws of this country make no provision for an official search as regards novelty, and all patents are taken out at the risk of the inventors. It is therefore incumbent on any person desiring to obtain a valid patent for an invention either to cause a search to be made, or himself to make a search as to the novelty of his invention. By omitting such a search, many a patentee has found, after paying his fees, that his treasured patent is worthless, because it has been anticipated. Of course, in this case the first applicant or patentee possesses all the Patent rights, and the second one has absolutely no rights at all.

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of printed Specifications of Patents is well over a quarter of a million.

A series of Indexes and Abridgments has been published by the Patent Office as a guide to the specifications themselves, and is freely distributed to the principal public libraries in this country. The Abridgments give a general description of the nature of every invention patented, and the object of their publication is to enable the would-be patentee to carry out, at any rate in some cases, what may be termed a *fireside search*. By the study of these Abridgments he will generally be able to select certain inventions which have already been patented, and which resemble his own invention sufficiently to render it desirable for him to examine their specifications in detail. A printed copy of any specification can be obtained at an inclusive price of 8d., through any Post Office, by a special Postcard (Patents Form C¹). The Abridgments are published in volumes, each volume dealing with one particular class of inventions, such as "Steam Engines," and "Cooking and Kitchen Appliances, &c." for a period of some years. The volumes up to 1877 are not illustrated, and all the subjects have not yet been dealt with, but from 1877 onwards a systematic series, very fully illustrated, is now in course of publication at a uniform price of one shilling per volume (including inland postage). The volumes for the periods from 1877 to 1883 and from 1884 to 1888 have been completed, those for the periods from 1889 to 1892 and from 1893 to 1896 are in active preparation, and later volumes will follow in due course. For the purposes of the Abridgments the whole field of invention has been divided into 146 "Abridgment Classes," and the list of these classes in itself shows what an enormous field this is, and how greatly its products vary. Every triumph of applied science, such as the locomotive, the telegraph, and the dynamo, is to be found here, and every one of our great national manufactures and industries finds its appointed place. Each volume contains abridged descriptions of the inventions falling under one of the 146 classes during the period of which it treats (illustrated by diagrams or drawings wherever possible), a detailed Index to the inventions according to their subject-matter, and an Index to the names of patentees or applicants.

For the use of those who desire to make a careful study of Patents, the Patent Office also publishes an "Abridgment-Class and Index Key" (price 1s., parcel postage 5d.), which shows in detail how inventions are classified, abridged, and indexed throughout its publications.

X Rays in Surgery. ("Razele X in Chirurgie.") By Prof. C. SEVEREANU. With 26 Radiographic Plates. Bucharest: F. Göbl. 1897. Pp. 94.

THIS latest contribution to our knowledge of X rays goes over, to a great extent, the same ground as the earlier books on the same subject.

In discussing the theory of the origin of the X rays, the author is of the opinion that they do not emanate from the cathode itself; that is to say, they are not cathode rays proper, but they are set up by the impinging of the cathode rays on some other body, either the glass envelope of the Crookes tube or the anti-cathode.

The illustrations are very good, and include a number of deformities, fractures, and freaks—such as a hand with six fingers, a foot with six toes, and such like.

The Chemical Composition of Waters. ("La Composicion Química de las Aguas." By JUAN J. J. KYLE, D.Sc., Professor of Chemistry at the University of Buenos Aires. Buenos Aires: 680 Calle Peru. 1897.

WE find in this pamphlet a large number of analyses of water from all parts of Brazil; some are ordinary potable

waters, but many contain a large quantity of dissolved salts. The results are expressed in parts per 100,000, and, though they represent a great amount of time and work, they are of no particular interest to English readers.

Manuali Hoepli.—*Manual for Chemists and Industrialists.* A Collection of Tables of Physical and Chemical Data, and of Processes of Technical Analysis. ("Manuale del Chimico e dell' Industriale." Raccolta de Tabelle, di Dati, Fisici e Chimici, e di Processi d'Analisi Technica ad uso di Chimici Analitici e Tecnici, &c.). By Dr. LUIGI GABBA. Second Edition, Enlarged and Enriched with the Analytical Tables of H. Will. Milan: Ulrico Hoepli. 1898. Pp. 442.

THE physical tables are extremely copious, and are satisfactory in every respect save the prominence awarded to the hydrometric scale of Baumé, which seems to take its stand on its essentially irrational character.

There is a useful table of molecular weights and solubilities in cold water, boiling water, and alcohol.

The fourth chapter, headed "Applied Chemistry," includes the chemical examination of potable waters. Here are given the chief components of the subsoil water of Milan; the regulations for the analysis of potable waters in Paris; directions for the assay of ores and metals, including pyrites, burnt ores, ores of copper, zinc, and lead; the electric assay of metals and alloys. Then follow assays concerning the heavy chemical industries, such as the analysis of the gases of pyrites kilns, and of chamber and tower gases.

According to Wedding & Hefner = to approximately $\frac{1}{2}$ Carcel, the luminous power of an Auer gas burner on and after 400 hours is the loss of about 25 per cent.

The assay of soaps, of wax, of mixtures of wax and tallow, and of spurious butters, is fully discussed.

Methods are given for determining the acidity of oils, for detecting cotton-seed oil, colza, sesame, and arachis.

After instructions for the examination of varnishes, we come to the assay of soaps, of textile fibres, tissues, and paper; and the analysis of soils, manures, &c.

The work is exceedingly rich in valuable and rare information. We can especially recommend it to the tropical and subtropical farmer and explorer.

Manuali Hoepli.—*La Grande Industria Chimica.* La Fabricazione dell' Acido Solforico et dell' Acido Nitrico del Solfato Sodico, dell' Acido Muratico. By Dott. V. VENDER, Chimico Consulente a Milano, formerly Chemist and Director of Water Works. With 107 Cuts and many Tables. Milan: Ulrico Hoepli. 1897. Pp. 279.

THE volume before us belongs to the useful series of Manuals now being produced by the firm of Hoepli, and treats of the most important branches of heavy chemical industry. The work opens with statistical generalities, which confirm the statement that among the chemical industries sulphur plays a part analogous to that held by iron in the mechanical world. In 1890 the world's production of sulphuric acid amounted to 2800 thousand tons, of which 870,000 were manufactured in Britain, 506,000 in Germany, and 234,000 in France. Italy contributes to the sum-total 120,000 tons. The American yield is not quoted. Among the various qualities of pyrites quoted, the highest grades are those obtained from Spain and Portugal.

The approximate table of atomic weights is, in one case at least, misleading, since it ranks platinum higher than gold.

The only objectionable feature in this work is that it persists in the exclusive use of the hydrometric or areometric scale of Baumé—distinguished for nothing but its disadvantages.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxv., No. 11, September 13, 1897.

Electrolytic Separation of Nickel and Cobalt from Iron. Application to the Determination of Nickel in Steels.—O. Durn.—Will be inserted in full.

Contributions to the Determination of Iron in Urine.—Dr. Adolf Jolles.—Hamburger's volumetric procedure has been modified so as to require less time. For the gravimetric determination nitroso- β -naphthol is recommended as a suitable reagent. The iron is separated from a hydrochloric solution by a concentrated solution of nitroso- β -naphthol in acetic acid at 50–52 per cent. The precipitate has the composition $C_{10}H_5O, NO_3Fe$.

Simplification of the Determination of Potassium.—Adolf Meyer.—The reagents necessary are normal barium chloride, normal oxalic acid, solution of platinum chloride containing 1 gm. Pt in 10 c.c. free from nitric acid, and lastly alcohol at 80 per cent by volume.

Iodine Number of Cacao Butter.—Dr. D. Held.—The value 51 is admittedly an error.

Determination of Boric Acid according to Gooch.—K. Kraut.—This paper requires the accompanying illustration.

No. 12, September 20.

On Oxycellulose.—L. Vignon.—Under the microscope oxycellulose appears to consist of very short filaments. It turns yellow at 100° and is insoluble in neutral reagents. It is turned blue by iodine and sulphuric acid. The colouration is more rapid and more distinct than with cellulose. The centesimal composition is—

Cellulose, Oxycellulose.			
C	44.44	43.55	
H	6.17	6.03	
O	49.39	50.42	

But if we submit oxycellulose to Lang's procedure (fusion with potassa at 180°) we find—

Cellulose, Oxycellulose.			
	P.c.	P.c.	
Soluble in fused KOH ..	12	87.58	
Insoluble	88	12.42	

We are led to consider this oxycellulose as a mixture of 75 per cent oxycellulose and 25 cellulose. The heat of combustion is as follows:—

Cellulose	4224 to 4190
Oxycellulose	4133 „ 4124

Absorption of Basic Colouring Matters.—Cellulose and oxycellulose have been compared from this point of view by steeping in baths of known strength, obtained with safranine and methylene blue, for thirty minutes at ebullition. Their impoverishment serves to measure the absorption. Schiff's reagent (magenta and sulphuric acid), prepared according to Villiers and Fayolle, yields, with oxycellulose, an intense violet colouration. It possesses, therefore, aldehydic functions.

On Retamine.—J. Battandier and Th. Malasse.—Retamine is capable of yielding neutral salts containing two mols. of monobasic acid or one mol. of bibasic acid to one mol. of alkaloid, and basic salts containing one mol. of monobasic acid to one mol. of alkaloid.

Influence of Colouring-matters upon the Fermentation of Highly-coloured Red Wines.—P. Carles and G. Rivière.—The incomplete transformation of sugar in highly-coloured musts is due to the colouring-matter, and

not to the acidity; for decoctions of elder, whether acidified or not, give the same results. This colouring-matter, allied to the tannins, acts as an antiseptic upon the microbes of fermentation.

No. 13, September 27.

Stability of the Phosphorescent Strontium Sulphides.—J. R. Mouret.—In general the phosphorescent strontium monosulphides are of little stability. Like the alkaline or alkaline-earthly sulphides, they have a tendency to become partially polysulphidised or sulphatised, forming hydrosulphates of sulphides. These properties, common to combination of sulphur with various metals, have no direct influence on the phosphorescence of strontium sulphide.

On Parastannyl Chloride.—R. Engel.—The facts summarised in this paper, and which will be developed in a subsequent memoir, readily explain the contradictions of former authors. The so-called *metastannic* acid, obtained at a low temperature, is in reality a mixture of stannic and metastannic acids.

On various Double Chlorides formed by Cinchonamine.—Léon Boutroux and P. Genvresse.—A knowledge of the facts observed by the authors permits us to avoid a cause of error in the detection of nitrates by cinchonamine.

Journal de Pharmacie et Chimie.
Series 6, vol vi., No. 4.

Histological and Chemical Study of the Action of Antiseptics on Muscular Fibres.—A. Riche.—This research is exclusively devoted to the action of antiseptics in the preservation of meat, in connection with a certain very dilute liquid; analysis has shown that it is formed of nearly pure bisulphide of lime. It is slightly acid, and gives off a slight smell of sulphurous acid; similar solutions of the same density were made from commercial sulphites of lime and soda. The matters examined were from the fillet of beef. Experiments were made by immersion in water, and in the reagents for seven hours, and the results compared. After immersion for thirty hours, the fibres were dissociated mechanically and examined under the microscope, and very important differences were found. Measurements of the diameters of the fibres were made, and there was found to be a remarkable difference between those treated in these three solutions and others treated with other reagents, such as boric acid, formol, &c.

Action of Iodine on Albumenoid Matters.—E. Lepinois.—The author considers that iodocasein might be susceptible of a therapeutic application in the case when thyroiodine and other iodised substances have been used or recommended.

On Aloines.—E. Léger.—Aloines have been derived from aloes from many parts of the world, but they may all be divided into two groups: the first comprises, according to various authorities, barbaloin, socaloine, zanaloine, and curacaloine; and the second only nataloin. This latter differs entirely from the others by its almost complete insolubility in water, even when warmed. It is also very slightly soluble in alcohol. According to some writers there appears to be no doubt that the first-mentioned four are identical.

Bulletin des Travaux de la Société de Pharmacie de Bordeaux. August, 1897.

Hygienic Value of Table Mustard.—P. Carles.—Not suitable for abstraction.

New Practical Apparatus for Lixiviation.—L. Barthe.—The apparatus proposed by M. Fonze-Diacon

is incontestably superior to that of Soxhlet, but it is too difficult and expensive to make and too fragile for general use. The author therefore recommends one that is very simple in construction and easy to use. A small tube, contracted at the lower end, is placed inside another of similar shape but larger, but they are prevented from touching closely by a thin glass rod which is placed between them: the large tube is fitted to a flask by means of a cork, and a vertical condenser is connected to its upper end; the vapour of the solvent passes through the interstices between the two tubes, and being condensed, falls down on to the substance to be lixiviated in the inner tube.

Coefficient of Partage of the Monobasic Fatty Acids of the Series $C_n H_{2n} O_2$ from the Condensation of C_1 to the Condensation of C_7 inclusive.—Th. Garraud.—A long paper, not suitable for abstraction.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xvii.-xviii., No. 15.

On the Oxidising Power of Manganous Salts, and on the Chemical Constitution of Laccase.—G. Bertrand.—Continuing his researches on this subject, the author finds that binoxide of manganese, stable in acidulated water, is reduced as soon as hydroquinone is added, giving a manganous salt, at the same time forming quinone. It follows from this that a definite weight of a manganous salt ought to oxidise, at the expense of the air, an unlimited amount of hydroquinone, or any other body equally oxidisable. The principal interest in the observations made in this paper is to complete, so far as concerns the chemical constitution and the mode of action of oxidases, an idea which sprung from the researches the author has already published, on the intervention of manganese in the oxidations provoked by laccase.

Existence of a Proteic Body foreseen by M. Bertrand in the Constitution of Oxidases.—J. de Rey-Pailhade.—The substance discovered by the author in 1889, and called by him "philothion," possesses the properties required by M. Bertrand.

On Cryoscopy of Milk and Organic Liquids.—A. Ponsot.—Two solutions having the same congealing point are not always equimolecular, and two solutions having the same point of congelation, under conditions of a given pressure, are in osmotic equilibrium under this pressure at the temperature of congelation; but they are not so as a rule at all temperatures, and in particular at the temperature of the organism.

On the Estimation of Caffeine in Coffee.—E. Tassilly.—The methods of estimating caffeine in coffee may be divided into three groups:—1. Exhaust with warm chloroform alone, in the presence of a base, such as lime, magnesia, ammonia. 2. Exhaust with warm water, and, after various treatments, the aqueous solution is agitated with chloroform, or evaporated with magnesia and treated by the same warm solvent in a digester. 3. The coffee alone or mixed with an alkali (lime or magnesia), is exhausted by a solution of an organic salt (benzoate or salicylate of soda), then the mass is exhausted by cold chloroform. These three methods are briefly described and criticised, and the author concludes that the methods of No. 1 group generally give but uncertain results; the methods by using benzoate or salicylate of soda give a very pure product, but it takes up too much time; but none of them are entirely satisfactory.

On a New Method for the Estimation of Caffeine in Coffee.—E. Tassilly.—The author proposes to treat the coffee with water, and then evaporate to dryness; the residue is then treated with sulphuric acid, and the caffeine dissolved in boiling water; at this point one can either evaporate to dryness in the presence of sand and

magnesia, followed by an exhaustion by warm chloroform in a digester, or, add ammonia and exhaust with cold chloroform in a flask.

On the Estimation of Oxide of Iron and Alumina in Phosphates.—N. Blattner and J. Brasseur.—Already inserted,

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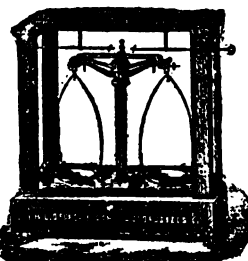
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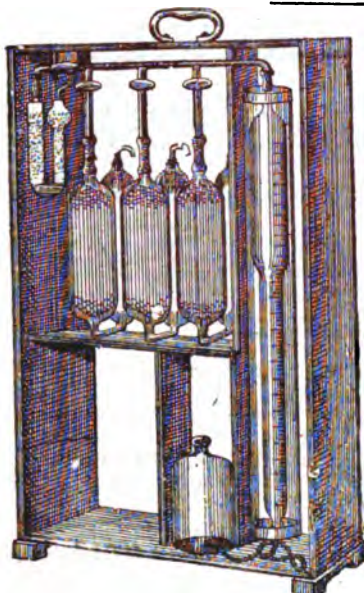
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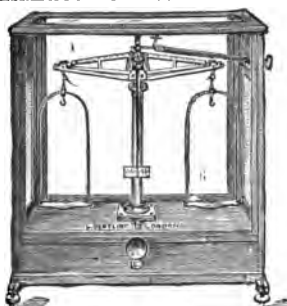
London: Printed and Published for the Proprietor by EDWIN JOHN DAVEY, at the Office, 6 & 7, Creed Lane, Ludgate Hill, E.C.
October 15, 1897.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1978.

THE PERMEABILITY OF ELEMENTS OF LOW ATOMIC WEIGHT TO THE RÖNTGEN RAYS.

By J. H. GLADSTONE, D.Sc., F.R.S., and
W. HIBBERT, F.I.C.

IN the CHEMICAL NEWS (vol. lxxiv., p. 235) there was published a condensed account of a communication made by us to the British Association, on the action of metals and their salts on the Röntgen rays. On December 18th (lxxiv., 298) appeared a longer paper, by Dr. Waddell, on the same subject; and in the CHEMICAL NEWS of October 1st of this year (lxxvi., 161) there appears a second paper which Dr. Waddell sent to Toronto. In this he attacks our general conclusions, specially with reference to the order of permeability of the alkaline metals lithium, sodium, and potassium.

While Dr. Waddell's knowledge of lithium is entirely derived from its salts, we determined its permeability in comparison with sodium and potassium from plates of the metals of known thickness. We thus got rid of any error due to the presence of the acid radicals.

Marangoni has repeated our experiments with larger quantities of the three metals, and come to the same conclusion as ourselves, namely, that lithium has an extremely small absorption. He finds that the metallic lithium must be ten times as thick as the sodium to produce the same absorption.

In judging the statements of Dr. Waddell and ourselves, it must be borne in mind that when we give the order of comparative permeability, we mean the permeability of an equal number of atoms, that is, equal atomic weights,—seven parts of lithium compared with twenty-three of sodium and thirty-nine of potassium, whereas Dr. Waddell compares equal absolute weights.

It is clear that in his method of observation the quantity of the acid radical cannot be the same in the salts which he compares. Yet the effect of this acid radical is very important. He does not seem to have perceived this at first. Even when further experiment gave him results which "surprised" him, he still retained in large part the opinions previously formed.

When the comparison is made for equal thicknesses of the three alkaline metals (not their salts), the order of permeability is still decidedly lithium, sodium, and potassium.

On attentively reading Dr. Waddell's last paper, we do not see that any one of his experiments is inconsistent with our conclusions, but in many cases our conclusions throw light on his difficulties.

The most original part of Dr. Waddell's paper is that on the peculiar granular appearance presented by the shadows of salts in his pill-boxes. We have frequently noticed the same phenomenon, but have never seriously investigated it.

We hope shortly to publish quantitative measurements of the intensity of some of these Röntgen ray shadows, a small number of which were in fact communicated to the British Association at Toronto.

Chemist to the London County Council.—At the recent meeting of the London County Council, the recommendation of the General Purposes Committee, that Dr. Frank Clowes, of Nottingham, be appointed successor to Mr. W. J. Dibdin, F.I.C., F.C.S., as Chemist and Superintending Gas Examiner, was unanimously confirmed.

FURTHER EXPERIMENTS ON THE LIQUEFACTION OF FLUORINE.

By M. MOISSAN and J. DEWAR.

IN May, 1897 (*Comptes Rendus*, cxxiv., 1202) we had the honour of presenting to the Academy our first experiments on the liquefaction of fluorine; we will now describe some fresh experiments we have made on this subject.

Liquefaction of Fluorine.—Our latest experiments on liquefaction were carried out by means of an apparatus similar to that which we have already described,—that is to say, a glass bulb fused to a platinum tube, which contained another similar smaller tube; but each of these tubes was fitted with a screw valve, in such a manner that, at any moment, communication—either with the outer air or with the current of fluorine—could be interrupted. This little apparatus was placed in a cylindrical glass receptacle with double sides, containing liquid air. The whole was connected with a vacuum pump, and furnished with a manometer.

In a series of preliminary experiments, we determined exactly the boiling-points of liquid oxygen at various pressures, as shown by the manometer.

In some former experiments we had shown that fluorine does not become liquid at the temperature of boiling oxygen at the ordinary atmospheric pressure.

We now find that, by repeating the same experiment with freshly prepared liquid air, the fluorine becomes liquid as soon as the air begins to boil, at the ordinary pressure.

We have repeated our former experiment, with liquid oxygen as refrigerant, and, on making a vacuum, we find that the liquefaction of fluorine takes place by the evaporation of the oxygen at a diminution of pressure of 32.5 c.m. of mercury.

From these two experiments we are enabled to state that the boiling temperature of fluorine is very close to -187° .

Experiments on Solidification.—When the little glass bulb was three-quarters full of liquid fluorine, we closed both the valves, and then caused the liquid air serving as refrigerant to boil rapidly, at a diminution of pressure of 72.5 c.m. Under these conditions we obtained a temperature of -210° . The fluorine did not show any sign of solidification, but retained its characteristic mobility. To complete this experiment it becomes necessary to cause the rapid ebullition of the liquid fluorine thus obtained; we hope to achieve this in future experiments. When we had repeated this experiment several times, a slight accident occurred to one of our little instruments containing the fluorine. The screw of one of the valves becoming worn, allowed the air to enter the bulb. This air was immediately liquefied, and in a few moments we had two distinct layers of liquid; the upper, colourless layer, consisted of liquid air; the lower one, of a pale yellow colour, being fluorine.

In another experiment, taking great precautions to prevent the ingress of any air, the fluorine was introduced in its liquid state into a glass tube, the end of which was then sealed before the blowpipe. The sealed tube, containing the liquid fluorine, was kept for a long time at -210° , by the rapid evaporation of a large quantity of liquid air, but it gave no trace of a solid body.

Density of Liquid Fluorine.—To determine the density of liquid fluorine, we brought it into contact with a number of bodies whose density is known exactly. By taking groups of bodies whose densities are very close to each other, it is easy to see which sink and which float in the liquid. This well-known though indirect method was the most suitable for these delicate experiments. We first of all satisfied ourselves that the fluorine had no action on the materials used. To effect this we placed a crystal of sulphocyanide of ammonium (density = 1.31) in a glass

tube surrounded with boiling liquid air; we then turned in, to the bottom of the tube, a current of fluorine gas, by means of a platinum jet. The fluorine was rapidly liquefied, and the sulphocyanide of ammonium was not attacked. We repeated the experiment with a fragment of ebonite ($D = 1.15$), of caoutchouc ($D = 0.99$), of wood ($D = 0.96$), of amber ($D = 1.14$), and of oxalate of methyl ($D = 1.15$). It is of importance, in the experiments we have just mentioned, that the various materials used should be first kept at a temperature of -200° , for some little time.

In one of our experiments a piece of caoutchouc, having been insufficiently cooled, took fire on the surface of the liquid, and burnt completely away with a brilliant flame, without leaving any residue of carbon.*

The experiment was carried out in the following manner:—In a glass tube closed at one end, and of which the lower part had been slightly drawn out, we placed fragments of the five substances we have just mentioned. The tube was then plunged to a third of its length into boiling liquid air. When it was all reduced to a temperature of about -200° we carefully introduced the fluorine gas. This soon became liquefied, and we saw the wood, the caoutchouc, and the ebonite floating easily on the surface of the pale yellow liquid. On the other hand, the oxalate of methyl remained at the bottom, while the amber rose and fell in the liquid, appearing to be of the same density. The apparatus was shaken several times, and the quantity of liquid fluorine increased, but the results were always the same.

We therefore arrive at the conclusion, from these experiments, that the density of liquid fluorine is 1.14. Another point which appears to be of interest is the following:—The fragment of amber floating in the fluorine was very difficult to distinguish, which would seem to indicate that the index of refraction of liquid fluorine is very close to that of solid bodies.

In another experiment we liquefied fluorine in a glass tube which had been previously graduated; we then sealed the tube, which had been weighed before the experiment, and left it alone in a beaker full of liquid air at the ordinary pressure. An hour and a half afterwards, the tube still being in 1 c.m. of liquid air, the fluorine had not changed in appearance. But shortly afterwards, when the air had all evaporated, a violent detonation occurred; the sealed tube and the double beaker in which it had been placed were smashed and reduced to powder. The sealed tube showed us that liquid fluorine sustains, at from -187° to -210° , a diminution of volume of $1/14$ th.

Absorption Spectrum.—We examined with the spectro-scope different samples of liquid fluorine through a thickness of about 1 c.m., either in sealed tubes or by means of our little condensing apparatus, but we have never been able to detect absorption-bands.

Magnetism.—Liquid fluorine placed between the poles of a powerful electro-magnet, does not show any magnetic phenomena. These experiments are the more decisive as we made comparative ones with liquid oxygen, as before; they were repeated several times.

Capillarity.—The capillary constant of fluorine is weaker than that of liquid oxygen. A capillary tube, plunged successively in fluorine, oxygen, alcohol, and water, gave the following figures:—

Height of liquid fluorine	3.5 m.m.
" " oxygen	5.0 "
" alcohol	14.0 "
" water	22.0 "

The Action of some Substances on Liquid Fluorine.

Hydrogen.—Liquid fluorine in a glass tube was cooled down by liquid air boiling at a low pressure. A slow current of hydrogen gas was made to impinge on the sur-

face of the yellow liquid by means of a platinum jet. There was immediate combination, with the production of a flame which lighted up the tube. The experiment was repeated by dipping the platinum jet below the surface of the liquid. At this temperature (-210°) complete combination still took place, with a considerable evolution of light and heat.

In another experiment the hydrogen apparatus terminated with a fine glass tube dipping into the liquid fluorine; when the hydrogen was turned on the combination took place immediately and with violence.

Oil of Turpentine.—Oil of turpentine, frozen and cooled down to -210° , is attacked by liquid fluorine. To perform this experiment we placed a little oil of turpentine at the bottom of a glass tube surrounded with boiling liquid air. As soon as a small quantity of fluorine was liquefied on the surface of the carbide the combination took place with explosive force, a brilliant flash of light, and deposition of carbon. After each explosion, the current of fluorine gas was kept up slowly, a fresh quantity of liquid fluorine was formed, and the detonations succeeded each other at intervals of from six to seven minutes. Finally, after a longer interval of about nine minutes, the quantity of fluorine formed was sufficient to cause, at the moment of the reaction, the complete destruction of the apparatus.*

Oxygen.—The action of liquid oxygen has been studied with much more care, since we observed from our earliest experiments that by passing a current of fluorine through liquid oxygen, we obtained a detonating body.

If we bring a current of fluorine on to the surface of liquid oxygen in a glass tube, the fluorine dissolves in all proportions, imparting a yellowish colour, and giving the liquid a graded tint from the upper to the lower part; the bottom of the tube is hardly coloured. If, on the contrary, we introduce the fluorine gas at the bottom of the liquid oxygen, the yellow colour is produced at the bottom and diffuses slowly to the upper layers.

This phenomenon indicates that the densities of liquid fluorine and oxygen are very near each other. When we have obtained a mixture of liquid oxygen and fluorine, if we allow the temperature to rise slowly, the oxygen evaporates first. The liquid becomes more and more concentrated as to fluorine, and finally the latter begins to boil in its turn. In fact, at the commencement of this boiling the gas coming off will light a match which has only a red-hot point, and will not make lamp-black or silicon red-hot; but, on the other hand, the gas coming off at the end of the experiment will instantly cause these two latter bodies to burst into flame. When the glass bulb is completely empty and its temperature is rising, we suddenly notice a distinct disengagement of heat, and the interior of the glass loses its polish. This rise of temperature is due to the fluorine gas attacking the glass. In this experiment, when using perfectly dry oxygen, no precipitate is produced. If, on the contrary, we use oxygen which has been some hours in contact with the air, the detonating substance we mentioned in our previous communication is produced with great readiness.

In one of our experiments, in which we tried to obtain a notable quantity of this body, we had an explosion strong enough to smash the glass in which the experiment was being performed.

To sum up, this body, which is produced by the action of fluorine on moist oxygen, seems to be hydrate of fluorine, decomposing, with detonation, by a simple rise of temperature.

Water.—We froze and cooled down to -210° a small quantity of water at the bottom of a glass tube. Liquid fluorine formed on the surface of the ice as a mobile liquid without action, and it evaporated on the temperature rising. As soon as the apparatus became warmer the remaining gaseous fluorine attacked the ice with great energy, and we noticed a strong smell of ozone.

* This piece of caoutchouc ran about the surface of the liquid like sodium on water, giving a very intense light.

* In several of our experiments we accidentally let a little liquid fluorine fall on the floor; the wood instantly took fire.

Mercury.—We solidified a globule of mercury at the bottom of a tube. The surface remaining very brilliant, the liquid fluorine surrounded it without causing it to lose its appearance or polish. On allowing the temperature to rise to -187° the fluorine began to boil, the liquid disappeared completely, but the attack of the mercury by the fluorine gas did not take place until the apparatus had almost reached the temperature of the laboratory.

Conclusions.

Fluorine gas is easily liquefied at the temperature of boiling atmospheric air. The boiling-point of liquid fluorine is -187° . It is soluble in all proportions in liquid oxygen and in liquid air. It does not solidify at -210° . Its density is 1.14, its capillarity is less than that of liquid oxygen; it has no absorption spectrum, and it is not magnetic.

Finally, at -210° it has no action on dry oxygen, water, or mercury, but it reacts, with incandescence, on hydrogen and oil of turpentine.—*Comptes Rendus*, cxxv., No. 15, p. 505, 1897.

NOTE ON THE ASSAY OF ELECTRO-PLATING AND GILDING SOLUTIONS.

By ALFRED H. ALLEN.

THE "Note on the Estimation of Silver in Silver-plating Solutions," by Mr. T. J. Baker (*CHEMICAL NEWS*, lxxvi., p. 167), appears to show that very imperfect methods of analysis for the assay of plating solutions are still in vogue. The method employed by Mr. Baker, namely, precipitation of the metals with cyanide by adding nitric acid, and cupellation of the precipitate, will no doubt give accurate results. It appears to me, however, to be less satisfactory on the whole than the following method devised and published by me some twenty years since:—

From 20 to 50 c.c. of the plating solution to be tested is largely diluted with water, and the liquid raised to the boiling-point. Sulphuretted hydrogen is then passed through the liquid, or ammonium sulphide gradually added. The silver falls as a black sulphide, which is liable to be contaminated with copper and zinc. The washed precipitate is rinsed off the filter into a flask or beaker, and treated with an excess of bromine water, which converts it rapidly and completely into argentic bromide. If any sulphur appears to have separated, a drop of bromine should be added to the residue, so as to ensure complete oxidation. Boiling water is now added, and the silver bromide is washed, dried, fused, and weighed. 188 parts by weight of the precipitate represent 108 of metallic silver.

For the determination of the precious metal contained in the solution of the double cyanide of gold and potassium used for *electro-gilding*, I have found the following method very satisfactory:—A measured quantity of the gilding solution is introduced into a porcelain crucible and cautiously concentrated. When in a syrupy condition, a few grms. of pure red-lead or litharge should be added, and the evaporation continued to complete dryness. There is little or no tendency to spitting. The crucible containing the residue is covered and raised for a short time to a moderate red-heat. The lead oxide is reduced by the cyanide present, with production of metallic lead and cyanate, and the reduced metal unites with the gold. The resultant button of metal is separated from the slag, and the gold contained in the alloy isolated either by cupellation or by treatment with pure nitric acid.

Electro-silvering solutions may be assayed in a precisely similar manner; but in this case treatment of the rich lead with nitric acid is, of course, inadmissible, and cupellation must be resorted to.

Sheffield, October 4, 1897.

APPARATUS FOR STUDENTS IN ELEMENTARY PRACTICAL CHEMISTRY.

By GEORGE GEORGE.

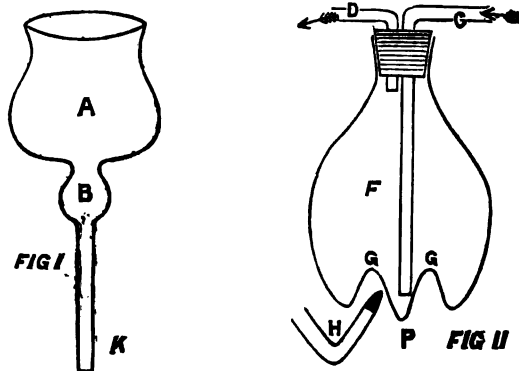
THE teaching of elementary chemistry has undergone a great change since 1889, when the Committee of the British Association made their Report upon the subject.

One of the outcomes of this progressive movement is that the student himself now performs a good many experiments which previously were shown only by the teacher. In consequence, the latter is constantly on the look-out for simple, effectual, and inexpensive apparatus, by means of which a large number of students can simultaneously establish for themselves well-known chemical truths or repeat chemical experiments.

The accompanying diagrams represent apparatus which I have found most satisfactory, and trust other teachers of chemistry may do likewise.

In Fig. 1 is shown a large thistle funnel, used for determining the equivalent weights of the metals, and by means of which boys—using a balance costing about thirty shillings—can obtain results involving less than 0.5 per cent of an error.

The funnel, which has a capacity of about 100 c.c., is made of thin glass, and weighs about 30 grms. B is a small bulb, in which asbestos wool is placed.



The *modus operandi* is as follows:—The bulb B is filled with asbestos wool that has previously been well washed with distilled water and dried. A short glass rod (about 2 inches), rounded at both ends, is then placed with the funnel, and the whole accurately weighed. Then, supposing it is required to find the weight of magnesium which will replace 100 grms. of silver, about 0.15 gm. of magnesium ribbon is weighed out and placed in the funnel A. A short piece of glass rod is fastened to the stem of the funnel at K, by means of a bit of rubber tubing. About 50 c.c. of a solution of silver nitrate (2 per cent) is now poured into A, and the magnesium ribbon well stirred with the glass rod, to remove the silver deposited upon it. After a short time the solution is allowed to run off, by removing the stopper at K. The stopper is then replaced, and a fresh quantity of solution placed in A.

When all the magnesium has been dissolved, the precipitated silver is washed repeatedly with distilled water, and finally with a little methylated spirits. The whole is then removed to the oven, dried, and weighed, the increase in weight representing the weight of the silver deposited.

Of course the same performance can be gone through with other metals and solutions, it being necessary, however, in some cases to heat the solution before placing it in A.

Fig. 2 needs little explanation. It represents a cross section of a flask designed to facilitate the collection of

water produced by a burning jet of hydrogen. The flask F is about 300 c.c. capacity, with rather a wide neck, and at the bottom drawn in all the way round as seen at G G', whilst a cone projects downwards and has its apex at F. Cold tap water is kept circulating in the flask by means of the tubes C and D, so that the cone F is always cold. The burning jet of hydrogen impinges upon this cone, and the water produced is there condensed, drips from F, and is caught in a vessel below. By means of this apparatus, practically the whole of the water produced by the burning hydrogen is collected, and in a short time sufficient can be obtained to determine boiling and melting points, density, &c.

Both of these pieces of apparatus can be made by the teacher himself, if he is at all expert in glass-blowing, or they can be obtained at a small cost from any glass-blower.

Allan Glen's School (Glasgow and W. of Scotland Technical College).

ELECTRICAL ENERGY CAUSED BY THE DIRECT ACTION OF THE ATMOSPHERE.

By H. N. WARREN, Principal, Liverpool Research Laboratory.

THE atmosphere, or, more correctly speaking, the gaseous constituent oxygen contained therein,—not only when in a combined form, as observed in most of the more energetic primary depolarisers, but even in the ordinary gaseous condition,—has, since the introduction of Sir W. Groves's well-known gas battery, been continuously experimented by various scientific men, in the hope of securing at least a somewhat economical supply of that subtle force now so largely employed over the entire field of civilisation.

Experiments of a lengthy nature which have also been engaged in, at the above laboratory, have at last terminated in the successful introduction of an electrical generator, which, even at the present time, although only in its infancy, is more than promising as a liberal source of electricity, not only on account of the extremely low rate of the chemicals employed, but also at the same time ranking next to perpetual motion on account of its perfect resuscitation, whereas the simplicity of its construction enables any one, without any special knowledge, to obtain at a trifling expense an electrical current of almost any required pressure.

The positive element of this powerful generator is composed of plates consisting of a special porous compressed graphite, of which one quarter of the plate is rendered active by immersion in platonic oxalate, and, after drying, igniting the same in an atmosphere of hydrogen gas. By this operation a very finely divided platinum surface is obtained, which, when in contact with a solution of ferrous sulphate, readily induces the oxygen of the atmosphere to combine with and to oxidise the iron present to the state of a ferric salt. In order to construct the cell, several of these so-prepared plates of graphite are attached to a circular lead beam, which surrounds a porous diaphragm containing as a negative element a rod of amalgamated zinc, the carbons being so arranged as to allow the platinised portion to project above the solution consisting of a strongly acidified portion of ferric sulphate. On completion of the circuit a powerful current is at once set free, and continues until the complete reduction of the ferric salt has taken place, which naturally terminates the action. On now withdrawing the zinc from the interior solution, an exactly reverse action is observed, the active platinum surface of the carbon condensing the atmospheric oxygen, which steadily re-oxidises the ferrous salt, and thus renews the action when required. With four ounces of ferric salt, contained in a generator 7 inches by 5, an electric current may be maintained with very slight fall at 2 v. 8 amp. for twenty-four hours.

A further modification of the above cell is now under experiment, in which it is hoped to be able to absorb the hydrogen gas by means of special manganese compounds, thus replacing the more expensive element platinum. For laboratories, and indeed any spot where a long-continued action is required, such as electro-dissolution, they compare very favourably with nitric acid cells, and all other depolarisers which evolve noxious vapours.

Liverpool Research Laboratory,
18, Albion Street, Everton, Liverpool.

DETERMINATION OF PHOSPHORUS IN STEEL, IRON, AND IRON ORES.

By JULIUS OHLY, Ph.D.

In the determination of phosphorus according to the molybdate method it has been customary either to obtain the yellow precipitate in the usual manner,—to dissolve it in ammonia water, reduce it by means of zinc, and titrate the ammoniacal solution with permanganate,—or to determine the amount of phosphorus present by means of a standard solution of sodium hydrate. In the first method the only indication for the completeness of the reduction has been found in the colour of the reduced liquid, which is supposed to be of an olive-green colour before the addition of the permanganate is admissible.

It has been found, however, that the operating chemist must be extremely careful and thoroughly experienced, in order to strike this point properly, and even then the results obtained have been found to be wanting. Besides the reducing operation with zinc, &c., is of a very tedious nature, and the tendency has been for these reasons to do away with the reduction of the ammoniacal solution entirely, and to substitute a more speedy and reliable procedure instead.

The following method is considered by the author as an improvement, as it does not leave anything to be desired in speed or reliability, being fully equal in results to those obtained by the gravimetric method.

Two grms. of sample (steel) are dissolved in 45 c.c. nitric acid, sp. gr. 1.16, in an 8-ounce Erlenmeyer flask, by heating until in solution. While hot, 5 c.c. saturated solution of potassium permanganate are added, and the whole allowed to boil until the pink colour has disappeared. Five or six drops of a saturated solution of sugar are then added, and, if these do not dissolve the precipitated oxide, a few drops more are added, avoiding carefully all excess. Let the flask cool now to about 60° C., add 5 c.c. ammonia, shake until clear, add 30 to 40 c.c. molybdate, shake well, allow to settle for a moment, filter with suction, wash with 2 per cent nitric acid six times, and rinse the flask with same. Then wash precipitate and flask with a 2 per cent solution of potassium nitrate, remove filter and precipitate from funnel, and put them back into the flask. Add 25 c.c. standard sodium hydrate solution, shake until the yellow precipitate is in solution, wash sides of flask down with water, add 3 or 4 drops of phenolphthalein, and titrate with standard nitric acid.

The standard nitric acid is made of such strength that 1 c.c. = 0.01 per cent phosphorus, i.e., 100 c.c. nitric acid (1.42) to 11 litres of water.

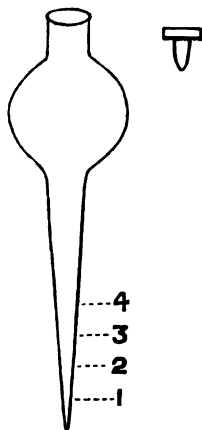
The nitric acid is standardised by running it against a steel whose phosphorus value has been determined gravimetrically.

The sodium hydrate may be of any strength; generally a solution is used of which 25 c.c. = 16 c.c. of the acid. A blank on the sodium hydrate ought to be run every day. The molybdate solution used is made by dissolving one pound of MoO₃ in 1200 c.c. water + 700 c.c. ammonia (0.90). The solution obtained is filtered, and 300 c.c. nitric acid (1.42) added. This forms the stock solution. To 1200 c.c. water and 475 c.c. nitric acid (1.42) add 575

c.c. of the stock molybdate, and use the solution thus obtained for determinations.

The method given above is used for pig-iron and ores with equal success. Unless there is a large percentage of insoluble carbon in the pig-iron it need not be filtered off. For ores it is necessary only that the phosphorus be all in solution in a nitric acid menstruum.

As the most important point to be reached by the chemist in these determinations consists in being enabled to give a reliable result in the possibly shortest time, another process, which gives very satisfactory results for all technical purposes, has also found introduction in some of the most prominent iron and steel works. It consists in the application of Goetz's phosphorus tubes, of the following shape:—



These tubes are charged with the properly prepared solution of iron or steel, as given above, the molybdate solution added, and are then placed into the circular openings of a disk which holds six or more of these filled tubes and rotates on a pivot. When charged it is set in motion by the application of the electric current, the latter giving to the mixture in the several tubes that motion which is generally given by the hand of the operating chemist. The scale, marked at the lower extremity of the tube 1, 2, 3, 4, tells directly the amount of phosphorus indicated by the bulk of the precipitate. The latter settles very rapidly in the lower part of the tube, and becomes very compact, and that to such an extent that the tube may be inverted without the operator incurring any risk of losing a part of the precipitate.

These tubes have been used for all technical purposes with great success for steel and iron exclusively, but there is no reason apparent why they should not be applied for the determination of phosphorus in ore as well.

DISSOCIATION SPECTRA OF SOME MELTED SALTS.

METALLOIDS; CHLORINE, BROMINE, IODINE.

By A. DE GRAMONT.

MOST melted salts give, under the action of a condenser spark of large capacity (which dissociates them), line-spectra in which each body is represented by the characteristic lines of its own spectrum (*Comptes Rendus*, July 8, 1895). I was by this means enabled to obtain some beautiful spectra of metalloids at the ordinary atmospheric pressure without having recourse to Plücker or Sait tubes.

I will now describe the spectra of chlorine, bromine,

and iodine, obtained by the same method, principally from their alkaline salts, in which the small number of metallic lines to deduct renders their study more easy.

In these experiments I used, for the purpose of holding the fused salts, two thick platinum wires, flattened out at the ends, arranged in V shape, the flattened ends forming the angle, the salts being then kept fused by means of a Bunsen. When the short spark is made to traverse the paste or melted salt between the wires the air lines disappear. The platinum lines rarely appear, and then only by accident, so long as a trace of the salt remains on the wire. I used a direct-vision spectroscopy with two compound prisms, constructed according to M. Cornu's design. It gives very good dispersion, but is rather absorbent. By removing one of the two systems of prisms, the entire spectrum could be examined, and even then the D line was distinctly doubled. That part of the spectrum between λ 700 and λ 430 was then measured over again with the two systems of prisms. The dispersion thus obtained gave for the lines D_1 and D_2 a separation on the micrometer which could easily be read to the tenth of a degree. The coil gave, without the condenser, and excited by four bichromate cells, a spark 50 m.m. long. The condenser was formed of Leyden jars, each surface of which was about 0.12 square metres, interposed in quantity in the secondary current. In these experiments I found it best to use from four to six of these jars, thus getting a condensing surface of from 0.46 to 0.70 square metres.

Condensers used in previous researches of this nature were much smaller in comparison with the coils used. We can now understand why, in the descriptions of metallic spectra produced from salts (notably Sir J. N. Lockyer, "Studies in Spectrum Analysis," 5th edition, Chap. VI., 1894), we find no mention of the appearance of the spectra of metalloids, which would, however, have been observed if a greater condensation had been used.

The wave-lengths here given were obtained by means of transformation curves of the readings of the scale of the spectroscopy. I made them for one and for two prisms by the aid of the figures given by M. Thalen for the principal lines of the metals, and more especially for those of iron. These numbers were compared with Angström's normal solar spectrum; to compare them with recent observations of the same photographic spectrum by Rowlands, we have only to refer to the table of corrections given in Appendix B of Watts's "Index of Spectra."

It is almost impossible to obtain in the ordinary work of a chemical laboratory a greater degree of accuracy than I have attained in the measurements of wave-lengths, even when giving, as I have, the averages of twenty readings in series of different observations.

If it is in the salts of the alkaline metals that one seeks the spectra of the metalloids, and this seems to be most probable, we should at first sight recognise the principal lines of potassium and sodium by simple comparison with Plate V. of M. Lecoq de Boisbaudran's beautiful Map of Luminous Spectra. We should also be able to study them with advantage in the alkaline carbonates; for, under the conditions of the experiment, these latter only give as carbon lines, and those very faintly, C α 658.3 and 657.8, in the neighbourhood of the red hydrogen line (the first hardly visible, even doubtful), and C β 426.6 in the indigo. It is, further, necessary to bear in mind the fact of the widening of the lines of the alkaline metals—the great condensation of the spark used makes this so diffuse as to almost completely efface the faint lines, leaving hardly any but the following lines visible:—

For sodium—

Na δ (616.0) (615.4), Na β (588.7) (588.2),
Na γ (497.9) double, Na α (589.5) (588.9),
Na ϵ (515.5) (515.2) diffused.

TABLE I.—Chlorine.

Chlorine (from melted chloride).		Free chlorine (in tubes).	
A. de Gramont.		Hasselberg.	Salet.
—	Not seen.	—	675 } A band divisible into four lines.
Cl α	610.5 Doubtful in the salts, T.	—	667 } Quite visible.
Cl β	1. 545.6 Well marked, T.	545.67	546.0 } Strong.
	2. 544.3 Fairly strong, T.	544.36	544.5 } Very strong.
	3. 542.3 " " T.	543.40	542.3 }
	4. 539.2 " " T.	539.24	538.9 }
532.5 Very faint, and of uncertain origin.		—	—
Cl γ	— Not seen.	528.47	—
	522.0 Strong, T. } First principal group. }	521.98	522.0 } Very strong; double.
	521.6 Strong, T. }	521.62	521.6 } The strongest.
	— Not seen. }	518.88	—
" } Seen by Thalén.		517.22	—
Cl δ	— " }	516.08	—
	1. 510.3 Fairly strong, T.	511.28	—
	2. 509.9 " " T.	510.24	510.2 } Double.
	507.7 Strong, T.	509.82	509.8 } Strong.
Cl ϵ	499.4 Fairly strong, T.	507.76	507.9 }
	497.3 Rather faint, T.	499.77	499.3 }
	— Not seen.	497.24	497.2 }
	— Not seen.	494.53	—
Cl ζ	1. 492.4 Faint.	493.79	—
	491.6 Very plain, T.	492.53	?
	2. 490.3 Fairly strong, T.	491.72	491.8 } Double.
	3. 489.7 " " T.	490.44	490.3 } Strong.
Cl η	1. 482.0 Very strong, T. } Second principal group {	489.69	489.6 } Strong.
	2. 481.0 " " T. }	481.98	482 } Strong.
	3. 479.4 " " T. }	480.97	481 } Strong.
	478.1 Well marked, T.	479.30	479.3 } Very strong.
Cl θ	476.8 " " T.	478.08	478 }
	474.0 Faint.	476.90	477 }
	457.2 Diffused; doubtful.	473.97	473.5 } Diffused.
	— Not seen.	—	457.8 } Band.
Cl ι	— " }	—	436 } Diffused.
	— " }	—	434.5 }
	— " }	—	432 } Diffused.
	— " }	—	431 }
Cl κ	— " }	—	427 } Band.
	— " }	—	425 }
	— " }	—	413.3 } Band.
	— " }	—	—

TABLE II.—Bromine.

Melted bromides (A. de Gramont).		Free bromine in tubes (Salet).	
—		700 approximately.	
—		678 " "	
—		663 " "	
—		658.2	
—		655.8	
—		654.5	
Br α	635.15 Well marked, T.	635.2	Fairly good.
Br β	614.6 Well seen, T.; appears single.	614.7	Small group; the widest line, and the least refrangible.
Br γ	587.0? Weak; uncertain.	587.5	
	582.95 Well marked, rather diffuse; appears single, T.	583	Double; the strongest and the most refrangible.
Br δ	571.9 Very weak, often hardly visible; appears single, T.	572.2	Double; the most refrangible.
Br ϵ	558.95 Well marked; appears single, T.	559	Double; the most refrangible; fairly strong.
	551.0 Well marked, T.	550.8	
	549.75 " " T.	549.6	Resoluble band.
	549.1 " " T.	549	
Br ζ	544.7 Weak, often hardly visible, T.	545	Resoluble band.
	543.5 Fairly good, T.	542.2	
	542.4 Good; double?, T.	532.6	Strong.
	1. 533.1 Very strong, T.	530.4	Fairly strong.
Br η	2. 530.4 Strong, T.	527.3	
	—	526.5	
	3. 523.65 Very strong, T.	524.0	Strong.
	4. 518.25 Very strong, T.	518.3	Strong.
Br θ	516.4 Well marked, T.	516.5	
	510.65 Fairly strong; variable; of doubtful origin; seen by Plücker.		
Br ι	505.4 Well marked, T.	506	

TABLE II.—*Bromine (continued).*

Melted bromides (A. de Gramont).		Free bromine in tubes (Salet).	
Br θ	492.9 Fairly strong, T.	493	Easily visible.
Br μ {	481.6 Very strong, T.	481.5	"
	478.6 " T.	478.7	Fairly strong.
	476.7 Easily visible, T.	476.6	"
	474.3 " T.	474.2	"
	472.0 " T.	472.0	"
Br ν	470.35 Very strong, T.	470.4	Fairly strong.
	469.15 Fairly visible; often weak.	467.6	"
	467.65 Fairly strong, T.	461.7	"
	462.2 Easily visible.	454.2	"
Br π	436.5 "	436.5	"
		428.7	"
		423 about	} Vague.
		418 "	
Br ρ		398	"

TABLE III.—*Iodine.*

Melted iodides (A. de Gramont).		Free iodine in tubes (Salet).	
I α	625.7 Fairly well marked.	625.7	"
	620.6 " "	621.0	"
I β {	612.6 Very well marked.	612.5	Easily seen.
	608.5 Fairly visible.	597.8	Double.
I γ {	607.8 Fairly strong.	595.2	Fairly strong.
	595.0 Strong.	579	"
	578.8 Fairly visible.	577.3	Easily seen.
	577.45 Well marked.	576	"
	576.0 Fairly well marked.	573.8	Fairly strong.
I δ {	573.75 Well marked.	571.1	"
	570.9 " "	568.8	Easily seen.
	569.1 Fairly well marked.	567.3	Fairly strong.
	567.75 Well marked.	562.4	"
	562.6 " "	561	"
I ϵ	561.3 Very faint.	559.6	"
	560.1 Faint (seen by Plücker).		
	559.5 Faint.		
	555.3 Very faint.		
	552.4 " "		
I ζ {	550.7 Faint.	550.1	"
	549.8 Strong.	549.4	Fairly strong.
	549.5 Fairly visible.	546.1	Strong.
	546.5 Very strong.	543.3	Easily seen.
	543.7 Strong.	540.3	Strong.
I η {	540.4 " "	536.6	"
	536.9 Fairly strong.	534.4	Fairly strong.
	534.3 Very strong.	533.6	"
	533.65 Very strong; almost coincides with Cd No. 3.		
	526.7 Easily seen.		
I θ	526.35 Fairly visible.	526.5	"
	524.45 Well marked.	524.3	"
	521.55 Well marked; almost coincides with Cl γ (521.6).	521.5	"
	517.55 Faint.		
I μ	516.15 Very strong.	516.2	Strong.
I ν	510.6 Well marked; variable.		
	506.5 Fairly visible.	506.5	"
I ξ {	486.4 Easily seen; diffused.	497.5	"
	484.96 " "	486.5	Easily seen.
	480.45 Faint.	485	Faint.
	476.5 " "	480.3	Very faint.
	473.15 " "	476.3	"
I $\omicron$ {	467.65 Well marked; almost coincides with Cd No. 6.	472.9	"
	466.7 Well marked.	467.7	Easily seen.
	464.15 " "	466.8	"
	463.1 " "	464	"
	462.2 " "	463.4	"
I π {	445.35 Fairly well seen; very diffused.	462	"
	444.9 Fairly visible; very diffused; often confounded with the previous one.	445.6	"
	443.9 Rather faint; diffused.	445	"
	441.1 Faint; diffused.	444	"
I σ	422.45 Very well seen.	441	"
		440	"
		442	"

And for potassium—

K δ (769.8) (766.5)	K β (536.0) (534.4) (534.0)
K γ (693.9) (691.1)	K ζ (482.8)
(630.8)	(426.4)
(611.7)	(418.5)
K α (583.2) (580.1) (578.3)	K ϵ (404.5)

The alphabetical designations which precede are from M. de Boisbaudran's map, and the figures are those which I obtained.

The spectra of free chlorine, bromine, and iodine are those which have up to the present been principally studied first by Plücker and later by Salet. The former made some excellent maps, but did not make any direct measurements of wave-lengths; these have, however, been deduced approximately by Watts, and will be found in his "Index of Spectra."

M. Salet then took up the question, and measured the wave-lengths obtained by a spark traversing a tube containing the free metalloid at ordinary pressure. He has since made some corrections in these figures, and I have reproduced them in this paper to facilitate comparison with those obtained by me.

For free chlorine only, more precise measurements than those of M. Salet have since been made by Thalén and Hasselberg, and I have thought it worth while to give M. Hasselberg's figures as being more recent. The lines marked T were observed also by Thalén. (See Table I.)

The following lines seen by Thalén in free chlorine were not perceived either by Salet, Hasselberg, nor by me in melted chlorides:—

559.35, 552.77, 535.55, 533.20, 531.25, 520.55, 517.40, 514.20, 503.05, 502.05, 477.35, 470.45, 469.80, 466.00, 464.80, 464.00, 463.80, 460.80, 459.60, 459.05, 452.70.

All the chlorides tried gave, with the greatest distinctness, the chlorine lines—with the exception, of course, of those which were effaced by their closeness to, or apparent coincidence with, the intense metallic lines. In a general way we may add that the entire spectrum of chlorine is much finer, the lines are brighter and sharper in the melted chlorides with a condenser spark than with free chlorine in tubes. The most characteristic groups of chlorine in the melted salts are principally Cl γ and Cl ζ , and, secondly, Cl β and Cl δ . The spectrum of chlorine has been examined in this manner in the melted salts of the chlorides of sodium, potassium, lithium, rubidium, cadmium, and zinc.

Bromine.

Since M. Salet's memoir, the emission spectrum of bromine has not, to my knowledge, been the object of any spectroscopic research; its spectrum, much richer in lines than that of chlorine, is as clear and as easily observed in the melted salts. Free bromine gave M. Salet a series of faint lines in the red, from 654 to 700, but I was not able to detect them in the bromides. The most characteristic groups of bromine in the melted salts are principally Br ν , Br μ , Br ζ_3 , ζ_4 ; and, secondly, Br ζ_1 , ζ_2 . The observations have been carried out with bromides of sodium, potassium, cadmium, and zinc. (See Table II.)

Iodine.

Like bromine, the emission spectra of iodine has not been made the subject of any research since that of M. Salet. It is richer in lines than are the spectra of either chlorine or bromine, notably in the orange red. The principal lines in this part of the spectrum, I α , I β , I γ , are easily seen, even with only a small quantity of iodine in the salt under examination. The most characteristic groups or lines of iodine in melted iodides are—Firstly, I γ , I μ , I ζ_2 ; and secondly, I ζ (as a whole), I ϵ , I α , I β , I γ . The experiments were performed on iodides of sodium, potassium, and cadmium.

The three spectra of chlorine, iodine, and bromine have their most characteristic lines in the green and the blue, and the complexity of these spectra, as well as their ex-

tension, is increased in the same manner as their atomic weights, and the sensitiveness of the observation appear to be in the same order. (See Table III.)

ON THE COMPOSITION OF CERTAIN CANADIAN VIRGIN SOILS.*

By FRANK T. SHUTT, M.A. F.I.C., F.C.S.,
Chemist, Dominion Experimental Farms.

(Continued from p. 186).

British Columbia.

BEGINNING on the west, or Pacific, coast, your attention is first directed to the tabular statement of the composition of certain typical British Columbian soils. (See Table I.)

These include three well marked groups:—

1. *Deltaic Soils*.—Very rich in plant food. These are formed by the accumulation of detritus, as at the mouths of the Fraser, Pitt, and other rivers.
2. *Valley Soils*.—Largely alluvial as regards origin; rich, as a rule, in both mineral constituent and organic matter.
3. *Benck and Plateau Soils*.—At varying altitudes on the sides and summits of elevations and mountains; variable, but usually light and sandy; of medium fertility, though sometimes very poor.

Possibly there may be other classes of soils in the province, but our investigation has as yet only included those now referred to.

Soil No. 1.—Taken from a valley near Victoria, Island of Vancouver, and representative of a large area that is considered good farming land. When air-dried, it is a dark brown, almost black loam, of excellent texture, homogeneous throughout, and containing clay and humus in good proportions.

In nitrogen and organic matter this soil ranks very high, and—though not as rich in total potash and phosphoric acid as many of our virgin soils—it is by no means deficient in these important constituents.

Soils Nos. 2 and 3.—Represent the soil immediately beneath the preceding sample at the depth of 12 to 18 inches and 18 to 24 inches respectively. In physical appearance and condition, as well as in composition, No. 2 is very similar to sample No. 1; showing that the surface soil has practically a depth of 18 inches. While, as might be expected, the lower sample (No. 3) is somewhat poorer in organic matter and nitrogen, the percentages of potash and phosphoric acid are identical with those in the overlying soil. It is of a yellowish grey colour with streaks of black soil throughout its mass. It will be seen to be of excellent quality for a subsoil.

It will be interesting now to consider the proportions or percentages of these elements that may be looked upon as more or less *immediately available* for plant use, i.e., the amounts extracted by the 1 per cent citric acid solution before referred to. (See Table II.)

In speaking of minimum limits of available plant food, Dr. Dyer says:—"From a careful consideration of the whole of the results, it would perhaps not be unreasonable to suggest that, when a soil is found to contain as little as about 0.01 per cent of phosphoric acid soluble in a 1 per cent solution of citric acid, it would be justifiable to assume that it stands in immediate need of phosphatic manure."

In potash he obtained results that led him to finally suggest that the limit to be regarded as indicating the non-necessity of the application of special potash fertilisers at 0.005 per cent of potash soluble in the solvent now spoken of.

* Read before the British Association (Section B), Toron Meeting, 1897.

TABLE I.—Analyses of Soils (Water-frees), British Columbia.

No.	Locality.	Surface or subsoil.	Character of soil.	Potash.	Phosphoric acid.	Nitrogen.	Lime.	Loss on ignition (organic and volatile matter).
1.	Victoria, Vancouver Island.. ..	Surface.. ..	Valley soil, black loam	0'23	0'19	0'594	1'29	15'69
2.	Victoria, Vancouver Island.. ..	Depth, 12—18 ins.		0'23	0'19	0'506	1'12	13'61
3.	Victoria, Vancouver Island.. ..	" 18—24 ins.		0'26	0'12	0'146	1'01	4'63
4.	Alberni, Vancouver Island.. ..	Surface.. ..	Dark red clay loam ..	0'32	0'08	0'127	1'14	10'79
5.	Alberni, Vancouver Island.. ..	"	Dark red sandy loam..	0'17	0'34	0'163	1'00	11'32
6.	Cowichan, Vancouver Island.. ..	"	Dark red sandy loam, Bench soil	0'39	0'32	0'102	1'37	7'10
7.	Ladners, New Westminster	"	Alluvial grey - black loam	0'52	0'28	0'610	0'50	17'25
8.	Squamish, New Westminster ..	"	Valley soil	0'38	0'20	0'091	1'68	3'38
9.	Pitt Meadows, New Westminster ..	"	Alluvial black loam ..	0'36	0'52	1'050	0'32	31'14
10.	Pitt Meadows, New Westminster ..	Subsoil.. ..	Greyish yellow sandy loam.. ..	0'45	0'13	0'095	0'33	6'37
11.	Agassiz, New Westminster	Surface.. ..	First Bench	0'32	0'24	0'159	0'86	6'87
12.	Agassiz, New Westminster	"	Second Bench	0'35	0'14	0'101	0'78	4'34
13.	Agassiz, New Westminster	"	Valley	0'39	0'18	0'154	0'96	6'92
14.	Agassiz, New Westminster	"	Valley	0'35	0'26	0'155	0'97	7'12
15.	Chilliwack, New Westminster ..	"	Valley soil, alluvial ..	0'63	0'21	0'166	0'98	7'72
16.	Chilliwack, New Westminster ..	Subsoil.. ..		0'51	0'23	0'108	0'90	5'90
17.	Mission, Yale ..	Surface.. ..	Light grey clay loam..	0'45	0'28	0'124	1'86	3'96
18.	" ..	Subsoil.. ..		0'62	0'33	0'076	1'90	3'35
19.	Guisachan, Yale ..	Surface.. ..	Light grey sandy loam	0'32	0'30	0'077	1'22	2'66
20.	" ..	"	Dark	0'53	0'30	0'236	1'70	6'18
21.	" ..	"	"	0'65	0'38	0'255	1'76	6'59
22.	" ..	"	"	0'55	0'34	0'259	1'25	7'13
23.	" ..	"	Light grey sandy loam	0'45	0'27	0'045	1'61	2'02
24.	Quesnelle, Cariboo ..	"	Dark grey sandy loam	0'39	0'22	0'399	17'77	12'01
25.	" ..	Subsoil.. ..		0'53	0'19	0'108	3'80	4'60
26.	Cottonwood River..	Surface.. ..	Yellowish sandy loam	0'32	0'34	0'234	1'14	8'28
27.	" ..	Subsoil.. ..	Very sandy.. ..	0'16	0'29	0'057	0'99	3'03
28.	" House.	Surface.. ..	Dark grey sandy loam	0'57	0'24	0'412	1'07	13'04
29.	" ..	Subsoil.. ..	Yellowish grey	0'47	0'10	0'050	1'22	3'02

TABLE II.—Comparison of "Available" with "Total" Amounts of Potash and Phosphoric Acid.

No.	Soil.	Potash.			Phosphoric acid.		
		Total potash.	Available potash.	Percentage of total potash available for plant use.	Total phosphoric acid.	Available phosphoric acid.	Percentage of total phosphoric available for plant use.
1.	Surface	0'23	0'00483	2'20	0'19	0'01020	5'66
2.	Between 12 and 18 inches	0'23	0'00299	1'36	0'19	0'01055	5'85
3.	Between 18 and 24 inches	0'26	0'00169	0'64	0'12	0'00588	4'90

In available mineral plant food the surface soil now under consideration is seen to give results approximating these limits. The estimations above tabulated are, however, more particularly useful in showing that the upper or surface portions of the soil contain much larger amounts of available food than the underlying soil. We are thus furnished with data to support the view that the greater productiveness of a surface soil, compared with its sub-soil, apart from the presence of nitrogen, in large part is due to the availability rather than to the total amounts of mineral fertilising constituents present.

Soil No. 4.—From Alberni, island of Vancouver; a clay loam of a deep red colour, masking entirely the presence

of the large amount of organic matter present. This sample is said to represent the soil to a depth of 9 inches over an approximate area of 10,000 acres. The sub-soil of this area is variable, sometimes clay, sometimes gravel and sand. In potash this soil (No. 4) is comparatively rich; in phosphoric acid, however, it is much below the average. As regards nitrogen it is of medium quality.

Soil No. 5.—Also from the district of Alberni, but differing from No. 4 in certain important features. It is known locally as "Fern and Sallal" soil, for the reason that on this virgin soil these plants grow most luxuriantly, crowding out to a great extent other vegetation. At first this soil gives but poor returns, but after several

ploughings—i. e., several seasons working—the yield increases, and good crops are obtained. An examination of the soil showed it to be distinctly acid to litmus paper. There is in this, no doubt, an indication of the cause of the infertility. The effect of exposure to the air through culture would be to correct this sourness, while at the same time locked-up plant food would be set free. Lime and wood ashes have given excellent returns on this soil.

The very large percentage of oxide of iron in these soils—exceeding, frequently, 20 per cent—is a feature worthy of note. It is probable that in the virgin soil a part of this iron is in the ferrous condition, due to the presence of organic matter and to certain other factors. The oxidising of this iron through cultural methods would free the soil of compounds injurious to the tender rootlets of agricultural crops. It is further important to point out that this soil, though yielding 1.0 per cent of lime to hydrochloric acid, sp. gr. 1.115, had a distinctly acid reaction, and was much benefitted by an application of lime.

Soil No. 6.—A Bench soil, deep red, of sandy character, from Cowichan, Island of Vancouver, similar in appearance to Nos. 4 and 5. It, however, contains less organic matter and nitrogen than these, but is not to be regarded as deficient in any of the essential elements.

A determination of the amounts of available potash and phosphoric acid, ascertained by the citric acid method, afforded the following data:—

Available potash		0.0089
Available phosphoric acid		0.0171

While these amounts do not fall below the limits named by Dr. Dyer, they are, however, such as to suggest that both potash and phosphoric acid would prove beneficial, and give good returns in increased crop yield.

Soil No. 7.—A greyish black soil of excellent texture, from the valley of the Fraser river near one of its mouths, and resulting from the deposition of silt brought down by this river. An area of over 30 square miles is, it is stated, covered by soil of this origin and character. Both from chemical and physical data, this soil would be judged an extremely fertile one, and practical results confirm this opinion. Of phosphoric acid, potash, and nitrogen it possesses quantities considerably above the averages already discussed for fertile soils.

Soil No. 8.—From the Squamish Valley, in the district of New Westminster. The valley is said to have an area of 14,000 acres of arable land. Its sub-soil is clay, though sometimes running into sand. Though containing adequate amounts of mineral food for crop requirements, it is below the average in nitrogen and humus. The ploughing under of green crops—preferably one of the legumes—has been found to improve this soil, both as regards tilth and productive power.

Soil No. 9.—From the Pitt Meadows, New Westminster. An alluvial deposit, composed of the detritus brought down by the Pitt River. It is black loam, in a moderately fine granular condition, and possessing a large amount of vegetable organic matter. On moistening it does not become plastic or sticky, and easily crumbles when dry. The soil granules display a remarkable homogeneity, proving the very intimate incorporation of the vegetable organic matter with the inorganic basis of the soil.

Its mechanical texture seems to be such as would allow freedom for root development, for permeation of air and percolation of water, while at the same time it is sufficiently compact and heavy to prevent easy leaching and to be retentive of moisture.

In potash and phosphoric acid it is seen to be well supplied, comparing most favourably in this respect with soils of great productiveness.

In nitrogen this soil is particularly rich, possessing about 34,000 lbs. per acre, estimating the weight of an acre of soil to the depth of 1 foot to be 3,500,000 lbs. The physical condition of this soil being such that nitrifi-

cation would proceed satisfactorily, the value of this large amount of organic nitrogen becomes obvious.

Soil No. 10 is the sub-soil of the above, and is a greyish yellow sandy loam. From its texture I should expect it to offer a very fair drainage to the surface soil.

Soils Nos. 11, 12, 13, and 14 are surface soils from the Experimental Farm at Agassiz. They are all of medium quality; in tilth rather light, and, though possessing a fair amount of clay, sand predominates. Though not presenting any marked differences, that of the first bench approaches closely in composition to that of the valley soil No. 14. The valley soils are, as a rule, distinctly richer than those occurring at higher elevations.

Soils Nos. 15 and 16 are from Chilliwack, on the Fraser River. They are valley soils, alluvial in origin. While not so rich as the delta soils of the Fraser and Pitt Rivers already discussed, they are by no means poor, possessing a good supply of potash and fair amounts of phosphoric acid and potash. They probably represent more or less truly the character of those soils of medium fertility found in British Columbia in many of her river valleys.

Soils Nos. 17 and 18.—A surface and sub-soil from Mission on Okanagan Lake, Yale district. Both are excellent as regards potash and phosphoric acid, but of poor tilth, caking on being dried into hard masses. The surface soil is somewhat deficient in organic matter, and might be much improved by drainage, judicious culture, and the turning under of a green crop—technically known as green manuring.

Soils Nos. 19, 20, 21, 22, and 23 are surface soils from the ranch of His Excellency the Governor-General at Guisachan. They are sandy loams of varying shades of grey, and, with the exception of Nos. 19 and 23, might be termed, as far as composition is concerned, soils of more than average fertility. These latter are, however, somewhat deficient in humus and nitrogen.

Soils Nos. 24 to 29 inclusive, are from plateaux and upper benches of the Fraser in the Cariboo district, a practically as yet unsettled area. Clover and indigenous grasses of good quality, it is stated, grow well upon them, and the probabilities are that the area here represented will be found suited for grazing purposes. Surface soils Nos. 24 and 28 are particularly rich, judging from the chemical analysis, and should prove very fertile if climatic conditions are favourable.

(To be continued).

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING SEPTEMBER 30TH, 1897.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolitan Water Act, 1871.

London, October 14th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 182 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from September 1st to September 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several

samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 182 samples examined during the month all were found to be clear, bright, and well filtered.

The rainfall at Oxford during September was 2.25 inches, the average for 30 years is 2.75 inches, making a deficiency of 0.5 inch. Rain fell on nine days only, but 1.13 inches fell on the 29th. The total excess for this year is now 1.12 inches.

In order to prevent any misapprehension in the public mind with regard to our monthly reports on the quality of the London waters, it may be advisable to repeat that the Water Companies in no way interfere with our position as absolutely independent scientific authorities. Further, they have no information antecedent to publication as to what will appear in our report. Our communications with the Companies are chiefly confined to calling their immediate attention to the least anomaly appearing in the character or the quality of the filtered water; our chief aim being to advise the engineers at the works as to the efficiency of storage and filtration.

There is no city in the world where such minute and incessant care is taken daily and almost hourly to detect and report on the slightest deviation from purity in its water supply.

The following table shows that the water supply of last month was actually in a higher state of purity than it was in the corresponding month of last year.

Comparison of the Averages of the Five Thames derived Supplies for the Months of September, 1896 and 1897.

	Common Salt.	Nitric Acid.	Hardness.	Oxygen reqd.	Organic Carbon.	Organic Carbon.	Colour.
	Per gall.	Per gall.	Degrees.	Per gall.	Per gall.	Per gall.	Br'n: Blue.
Means.	Means.	Means.	Means.	Means.	Means.	Max.	Means.
Sept, 1896.	2.228	0.834	13.41	0.048	0.087	0.146	14.0: 20
1897.	2.137	0.836	14.25	0.034	0.087	0.104	11.1: 20

Our bacteriological examination of 255 samples taken by us have given the following results; we have also examined 9 other samples, from special points, making a total of 264 in all:—

	Microbes per c.c.
Thames water, unfiltered (mean of 26 samples)	98,485
Thames water, from the clear water wells of five Thames-derived supplies (mean of 126 samples)	72
Ditto ditto highest	992
Ditto ditto lowest	2
New River, unfiltered (mean of 26 samples)	387
New River, filtered (mean of 26 samples)	26
River Lea, unfiltered (mean of 26 samples)	3168
River Lea, from the clear water well of the East London Water Company (mean of 26 samples)	36

Apart from the daily bacteriological examination of the clear water wells of the Companies, we frequently make specific tests for the presence of pathogenic organisms. If any other than a negative result had been obtained the fact would have been recorded.

It will be observed in the above table that there was an occasion in which the microbes rose to far above the average. This was quite exceptional, and was connected with the atmospheric conditions following an unusually violent thunderstorm, when 1.13 inch of rain fell in less than an hour. We are in a position to say these microbes were harmless.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.

JAMES DEWAR.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 14, October 4, 1897.

On Antique Glass Mirrors Lined with Metal.—M. Berthelot.—Such metallic mirrors are mentioned by Pliny as manufactured at Brinduvium with an alloy of tin, in which I find the origin of the word *bronze*, which has been so long uncertain.

New Method for the Assay of Metals.—A. Frémont.—The points to be determined are merely the tenacity, the ductility, the fragility, and the homogeneity of the metal in question, and the procedure employed is of course purely mechanical.

On the Photographic "Veil" in Radiography.—V. Chabaud.—The author concludes that—(1). Setting out from a given resistance, the two electrodes of the tube emit alternately cathodic rays, and consequently create two foci. (2). Setting out from the same resistance, the tube emits X rays in all directions; in fact, those rays which take their rise on the second focus do not encounter any obstacle in the tube, and propagate themselves in all directions. (3). A hard tube will require a shorter exposure than a soft tube, but will yield proofs more veiled and less distinct than those furnished by the latter. (4). A voluminous tube with large electrodes will give on the screen a greater luminosity than a tube of small dimensions and small electrodes, but the former gives a less definite image.

Solubility of Liquids.—A. Aignon and E. Duzes.—Not suited for useful abstraction.

Journal de Pharmacie et Chimie.

Series 6, vol vi., No. 5.

Action of Sulphuric Acid on Levo-turpentine.—G. Bouchardat and J. Lafont.—The authors incorporated with essence of French levo-turpentine a tenth part of its weight of sulphuric acid; the product was heated to 150° with excess of potash-alcohol; this was treated with plenty of water. The supernatant oils consist of unchanged turpentine, a little camphene, terpenes, and their liquid polymers boiling at 310° to 320°, and of others solid at a low temperature. The water retains in solution the potassic salts formed by this reaction. These salts were first re-crystallised in water, to eliminate the large excess of alkali, and then in concentrated alcohol. They act on polarised light; they consist of two distinct bodies of the same chemical composition, and have, after repeated crystallisations, been separated. The least soluble of these salts, which we call levo-terebenthenosulphate of potash, is in the form of lamellar crystals, similar to boric acid; its rotatory power in 50 per cent alcohol is $[\alpha]_D = -25^\circ$. The second salt is found in the form of long, felted, silky, anhydrous needles, but its rotatory power is different; under the same conditions of dilution we get $[\alpha]_D = +10^\circ$. The mother-liquors of the preceding salts contain two other saline compounds of a different nature; we are now investigating them.

Composition of Haricots, Lentils, and Peas.—M. Balland.

No. 6.

Estimation of Lime, Alumina, and Iron in Mineral Phosphates.—L. Lindet.—Will be inserted in full.

On the Question of Matches, Phosphorism.—A. Riche.—A long, interesting, historical paper, to be continued, but unsuitable for abstraction.

MEETINGS FOR THE WEEK.

FRIDAY, 29th.—Physical, 5. Professor Stroud will exhibit and describe the Barr and Stroud Naval Range Finder. "A Telemetrical Focometer and Spherometer," by Prof. Stroud. Mr. Ackermann will exhibit a Surface Tension Experiment.

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EXPLANATION.

THE foregoing offer has been published in *Nature* and in the *CHEMICAL NEWS*. It has therefore presumably been brought to the notice of students of science in all parts of the world.

In order to give reasonable time for research and for distant communication, a period of six months will be allowed for the sending in of competing papers. That is to say, all papers intended for competition must be received by the Argentaurem Syndicate not later than April 30, 1898.

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No limit, either maximum or minimum, is assigned to the length of any of the papers.

Immediately after April 30th, 1898, the papers will be read and compared; and the adjudication will be announced by advertisement in *Nature* and the *CHEMICAL NEWS* during July, 1898.

The right of publication and copyright of the prize papers will belong to the Argentaurem Syndicate without any payment, other than the prizes, to the respective authors.

Unsuccessful papers will be returned to the writers; but in cases where such documents may contain information and observations of value as supplementary to the prize papers, the Argentaurem Syndicate will offer to purchase the papers in question.

The prize papers (and supplements, if any) will be issued in book form by the Plain Citizen Publishing Company, and a copy will be presented free to every competitor.

The sole judge of the competition will be Dr. Stephen H. Emmens, the President of the Argentaurem Syndicate. This is the only practical course. If, however, after the publication of the successful papers, any competitor should consider his own work as being more meritorious than the corresponding prize-paper, he will be allowed to submit the case to three arbitrators (one chosen by himself, one by the Argentaurem Syndicate, and the third by the two thus selected); and if the verdict of this tribunal be in his favour the Argentaurem Syndicate will pay an extra prize of \$500 for each paper so distinguished.

Apart from this provision for absolute fairness of adjudication, reference is made to *Argentaurem Papers No. 1* as showing that Dr. Emmens is duly qualified to act as judge of the competition. That book has been before the scientific world since January of the present year, and although it calls attention to some serious defects and errors in current doctrines, it has remained without serious impeachment. It may, indeed, be fairly said to be unimpeachable; for no claimants came forward

to take the prizes amounting to \$10,000 offered for the discovery of scientific errors in its pages.

The Argentaurem Syndicate has two objects in view in making the present offer.

In the first place, it is desired to gather together and make generally available the scattered items of Knowledge that exist with regard to the subjects propounded. That this is needed may be understood from the pitiable exhibitions of ignorance that constantly appear even in professedly scientific journals and in the utterances of professed scientific experts.

In the second place, it is desired to aid the Luthers of to-day in their protestant movement against Scientific Papacy. So far as truth-seeking and right-doing are concerned, there is nothing to choose between Official Politics, Official Religion, and Official Science. The doctrine of "my country, right or wrong" has its counterpart in "my doxy, right or wrong," and in "my dictum, right or wrong." A Cabinet will deliberately deceive a nation; a Church will deliberately make heresy a crime; and a Learned Society will deliberately excommunicate Truth. History abounds in instances of such proceedings.

Professorships, fellowships, and memberships are the passports to repute and livelihood in the world of science, and they are in the gift of certain presiding conclaves. It is no matter of wonder, therefore, to find individual men of science dumb in the presence of a Luther, and their papal authorities agreed upon the expediency of his being strictly boycotted. Fortunately, however, for the cause of true Science as distinguished from Doctrine, the combustion and physical restraint of heretics are no longer practicable; and if the Luther be a man of determination and pecuniary means he can make his protest plain to the world at large, even should the popes use against him their most powerful weapon, namely, a conspiracy of silence.

The offer now made by the Argentaurem Syndicate will, it is hoped, result in the downfall of various unsound doctrines at present taught by many a Fellow of the Royal Society and by many a member of the Académie des Sciences. Some of these doctrines are so childishly absurd as to be a reproach to the intelligence of the age; yet, strange to say, men of high intellect and real eminence like Lord Kelvin, Prof. Tait, Sir R. S. Ball, Prof. Newcomb, and Prof. C. A. Young, go on teaching them from year to year, and insist upon the students they instruct remaining blind to the light that is leading to progress in the outer world.

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Edited by
Sir Wm. Crookes, F.R.S.] (WITH WHICH IS INCORPORATED THE "CHEMICAL GAZETTE")

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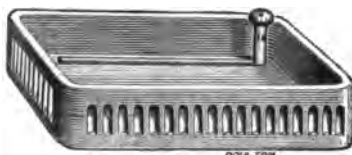
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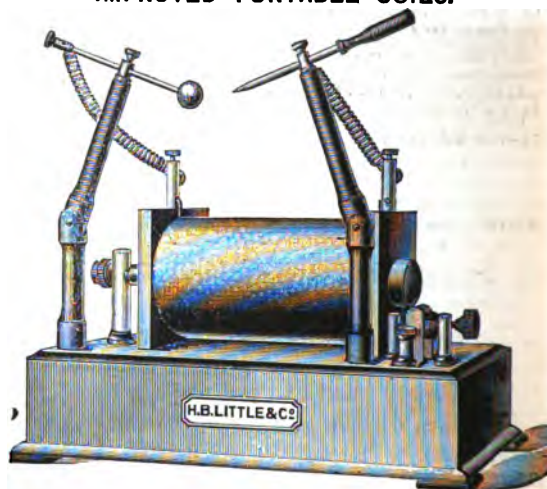
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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1979.

THE DISTRIBUTION OF CARBONIC ACID IN THE AIR.*

By W. CARLETON WILLIAMS.

It is well known that the method of estimating carbonic acid in the air, as originally proposed by Pettenkofer, contains a source of error arising from the action of oxalic acid on barium carbonate. Another source of error, due to the action of the baryta on the walls of the glass cylinder, in which the experiment is conducted, has been pointed out by W. Spring (*Mém. Acad. Royale de Belgique*, vol. xxxvii.), and more recently by Letts and Blake (*Chem. Soc. Proc.*, 1896, p. 192).

A series of determinations made by a modification of Pettenkofer's method, in which these inaccuracies were avoided, gave the following results for air collected in the suburbs of Sheffield, one and a half miles W.S.W. of the centre of the town.

	Number of experiments.	Average.	Minimum.	Maximum.
Suburbs	142	3'26	2'16	5'14
Fog	7	3'94	—	—
No fog	135	3'24	—	—
Snow	32	3'58	—	—
No snow	110	3'24	—	—
Rain	—	3'12	—	—
Fine	—	3'14	—	—
Centre of the town	21	3'85	2'80	6'22

The carbonic acid is distinctly higher in the town than in the suburbs. A marked increase is produced by fog

* Abstract of paper communicated to the Commemorative Volume of the University College, Sheffield.

and snow; rain does not exert any decided influence. The observations were made during the months of December to April, and, as far as they go, the results show a maximum of CO₂ in January (3'65), with a steady fall to April (2'64). The carbonic acid diminishes as the temperature rises. This is probably accounted for by the decrease in the consumption of fuel for household purposes during the warmer weather. An increase in the amount of carbonic acid is observed with a very high or very low pressure of the atmosphere.

A difference of opinion appears to exist with reference to the question of the uniform distribution of the carbonic acid in the air of inhabited buildings. In the well-known "Handbook of Hygiene" by Parkes it is stated, on the authority of Lassaigue, Pettenkofer, and Roscoe, that the carbonic acid of respiration is equally diffused through the air of a room. Angus Smith, on the other hand, asserts ("Air and Rain," p. 68) that "the air at the ceiling is generally the worst. This, however, depends upon circumstances; if it has time to cool from the height and space being great, the carbonic acid may be arrested before reaching a great height."

Every-day experience would lead us to suppose that Angus Smith is right: our sense of smell indicates that the air near the ceiling of a room is, as a rule, appreciably less pure than the air four or five feet above the floor.

In order to decide between these conflicting statements, samples of air collected in public rooms, class-rooms in Firth College, and dwelling-rooms in private houses, were examined. The results clearly show that Angus Smith is correct. In lofty rooms, from 26 to 31 feet in height, the air near the ceiling contained less carbonic acid than the air near the floor, but in rooms of a height between 9½ and 16 feet, the air collected 2 feet below the ceiling invariably contained more carbonic acid than the air taken at 2 feet above the floor. As is naturally to be expected, the difference is greater at night when gas or lamps are burning than it is in the day time. During the day, the mean difference is 1·7 volumes in 10,000, but at night the air at the top of the room contained, on an average, 3·7 volumes of CO₂ in 10,000 in excess of that present in the air 2 feet above the floor. These results show that the distribution of CO₂ in the air of an inhabited room is not uniform. (See accompanying Table).

	Height. Ft. Ins.	Space. In cubic feet.		° C.	Vols. CO ₂ in 10,000.	
Small class-room	16 0	2656	Ceiling, 11.30 a.m.	17	5'19	7 persons.
" " " " " "	" "	" "	Floor, " "	16	5'15	Gas-stove burning.
" " " " " "	" "	" "	Ceiling, 3 p.m.	17	10'48	4 persons.
" " " " " "	" "	" "	Floor, " "	16	10'23	Gas-stove burning.
PRIVATE HOUSE.						
Bedroom	11 6	3812	Ceiling, 8 a.m.	14'5	4'15	2 persons.
" " " " " "	" "	" "	Floor, " "	14	3'51	
Dwelling-room A	" "	" "	Ceiling, 9 p.m.	15	11'28	
" " " " " "	" "	" "	Floor, " "	13	9'91	
" " " " " "	" "	" "	Ceiling, 3 p.m.	17'5	7'09	Open fire; 3 persons.
" " " " " "	" "	" "	Floor, " "	17'25	5'27	
" " " " " "	" "	" "	Ceiling, 9 p.m.	19	16'66	Open fire; 3 people.
" " " " " "	" "	" "	Floor, " "	16	10'31	2 gas jets; 1 oil lamp.
" " " " " "	" "	" "	Ceiling, 8 p.m.	18'2	13'08	
" " " " " "	" "	" "	Floor, " "	13'5	11'28	
" " " " " "	" "	" "	Ceiling, " "	20'5	10'45	3 persons in the room.
" " " " " "	" "	" "	Floor, " "	18	7'63	Open fire, lamp, and candles.
" " " " " "	" "	" "	Ceiling, 4 p.m.	15	5'59	Open fire; 3 people.
" " " " " "	" "	" "	Floor, " "	14	5'04	
" " " " " "	" "	" "	Ceiling, " "	16'4	5'31	Open fire; 1 person.
" " " " " "	" "	" "	Floor, " "	13'6	3'31	
" " " " " "	" "	" "	Ceiling, 8 p.m.	18'4	10'47	Open fire; 1 lamp.
" " " " " "	" "	" "	Floor, " "	14'6	5'07	1 gas jet; 1 person present.
" " " " " "	" "	" "	Ceiling, 6 p.m.	14'4	6'62	
" " " " " "	" "	" "	Floor, " "	12'5	4'18	Open fire; 2 people.
" " " " " "	" "	" "	Ceiling, 7.40 p.m.	18	10'48	Open fire; 3 people present.
" " " " " "	" "	" "	Floor, " "	13'8	5'73	2 gas jets burning.

SEPARATIONS WITH ALKALINE ACETATES.

By HARRY BREARLEY.

(Continued from p. 177).

V. Aluminium and Copper from Iron.

A SOLUTION of aluminium chloride containing considerable free acetic acid cannot be precipitated by an excess of alkaline acetate; and precipitates formed locally are dissolved on immediate stirring, although if such a precipitate is allowed to roll about unbroken it becomes difficultly soluble.

Qualitative tests made some months ago induced one to believe that, as in the case of chromic salts, a very considerable separation of aluminium from iron might be made in presence of large amounts of free acetic acid.

A very short experience, under well-defined conditions showed an altogether unexpected difficulty. An example will readily illustrate the point. Say that a solution containing 1 gm. iron, 0.1 gm. Al, its maximum amount of dissolved hydrate, 10 c.c. acetic acid, and 10 to 12 c.c. acetate, is heated to boiling. A similar solution, lacking only the aluminium, would have its iron precipitated during the operation; but the sample would not show the least trace of turbidity. An additional 10 c.c. of acetate would form a local precipitate, which would be readily dissolved as it moved through the liquid. With about 40 c.c. of acetate the solution would become turbid, but if the boiling were continued for an hour or so there would be no considerable separation of iron. Indeed it would need about 60 c.c. of acetate to separate the iron so far as to produce a colourless filtrate. If the deep-coloured supernatant solution of a partial precipitation is poured on to a large asbestos disc, the filtration is sharply arrested in a manner very suggestive of alumina.

This peculiarity, in a similar degree, is noticeable whether the acetic acid be increased or decreased, and whether the dissolved hydrate be as stated or none whatever. These observations show that the practice, in co-precipitating iron and aluminium, of adding considerably more than enough acetate to precipitate the iron, is a necessary one; they also take the edge off one's surprise at finding only a small percentage of aluminium separated by any modification of the acetate separation.

Any useful separation of Al and Fe by means of alkaline acetates appears to be hopeless. On this account the alumina separated from the filtrate was never quantitatively estimated.

It is only necessary now to remark that, in the presence of large quantities of aluminium, some of the separations previously noticed may not be performed with perfect accuracy. To precipitate 1 gm. of iron, after neutralising and adding 10 c.c. acetic acid, would require—

In presence of 0.1 gm. Al 60 c.c. acetate.

"	0.05	"	35	"
"	0.01	"	20	"
"	0.0025	"	14	"

Luckily the proportion of Al in steel usually falls near the last-named amount, and would therefore occasion no appreciable change. For the sake of completeness, however, it is desirable to enquire how the 60 c.c. of acetate would act in a nickel estimation (say) if as much as 10 per cent aluminium chanced to be present. Two tests, with 0.1 gm. of Ni present, yielded percentage recoveries of 98.0 and 98.4 respectively. A reference to Table XIII. (p. 166) will show that this is a much better result than is obtainable with pure mixtures of iron and nickel. If it be not too great a liberty to say so, it seems as though the additional acetate was too busily engaged with the aluminium to give much attention to the nickel.

A more complete separation of iron and nickel, under like circumstances, might be made by adding large amounts of ammonium chloride. With this modification less acetate would be needed, and, on the authority of

Jewett (CHEMICAL NEWS, xl., 273) a more perfect separation is ensured.

Assuming a perfect separation of Fe and Ni, a substitution of alkaline chromate for acetate would readily precipitate the iron, but a very considerable—how much has yet to be determined—amount of aluminium is to be found in the filtrate. When precipitated as alumina in the subsequent titration, this element would be highly objectionable, not only on account of its interference with the indicator, but also on account of its possible influence on the titration itself.

According to Moore (CHEMICAL NEWS, lxxii., 92), "in presence of alumina, either citric acid, tartaric acid, or soda pyrophosphate, may be employed to keep it in solution." The points raised in these two paragraphs will be considered in a later paper.

Copper from Iron.

The acetate separation of copper cannot be said to have ever been greatly favoured, partly, it may be surmised, because a casual separation would almost certainly be a bad one. Wherever the separation is now used, it is generally accompanied by the volumetric estimation of the copper, without a preliminary separation of the precipitated iron.

The possibilities of acetate separation will be best understood by comparing its behaviour when an excess of acetate is used with one of the previously separated elements under like conditions.

Table XV. sets forth this comparison. The corresponding precipitations were made under as identical conditions as could be maintained in two sets of experiments made so widely apart. Attention is again called to the turbidity temperatures as evidence of the similarity. What difference there is favours the copper.

TABLE XV.

Acetate. Cc.	Percentage recovery of		Respective Temp. turbidities.
	Nickel.	Copper.	
10	100.0	98.0	— 91° C.
20	99.0	92.9	72°, 74° C.
50	95.2	69.6	60°, 62° C.
100	90.0	53.1	53°, 54° C.

Like most of the preceding elements, the separation is increasingly accurate as the free acid rises, and decreasingly so as the acetate rises. Any attempt to increase the volume of acetic acid necessarily entails an increase of the acetate required to effect a separation. So far as copper is concerned this is a disadvantage in that a less perfect separation is obtainable with a minimum acetate, and somewhat of an advantage in that a given excess of acetate causes a slighter decrease in the recovery. This advantage or disadvantage is probably true of nickel, cobalt, manganese, and every other element—except chromium as chromic acid—whose acetate separation from iron is practicable, although none of the preceding metals have been nearly so sensitive as is copper in this respect.

TABLE XVI.—With 30 c.c. Acetic Acid.

Acetate.	Per cent recovery.	Turb. temps.
30 c.c.	95.2	93° C.
50 "	91.2	84° C.

It might be presumed, from the foregoing, that a smaller volume of acid (acetic) with only so much acetate as would precipitate the iron after prolonged boiling would give improved results.

Two samples were boiled—

Minutes.	Per cent recovery.
18	98.6
40	98.8

The filtrate of the first sample was distinctly tinted with

unprecipitated iron. Evidently mere prolonged boiling has no effect on the separation.

To go a step further, and passing over decreasing volumes of acid and acetate, which would give increasingly better results, it might be predicted that Schwarzenberg's or Herschell's method, which consists of boiling a solution containing the maximum amount of dissolved hydrate, would give the best results of all.

Truly it might be objected that this would not be an acetate separation, nor would it, although in a mathematical sense it may be regarded as the limiting case of such separations. In a practical sense, too, it may often be convenient to convert a Schwarzenberg into an acetate separation, when the neutralisation has not been performed with the necessary exactness. The Schwarzenberg reaction is generally assured by adding large amounts of alkaline chlorides. In the two cases given below no such addition was made. In neither case was the solution filterable when the boiling point was reached. I. was boiled five minutes; II. thirty minutes.

TABLE XVII.

	Temp. turb.	Per cent recovery.
I.	81° C.	99.45
II.	83° C.	99.10

These results are particularly noteworthy, because copper is not one of the metals where separation from iron by these means is generally recommended.

The preceding tests were all made with soda salts, so as to accommodate the filtrate to the modified cyanide estimation of copper, an account of which is to be found on page 189.

The presence of soda chloride in unusual quantities rather improves the separation.

Separations made with ammonia salts yield somewhat better results, under like conditions, than do soda salts. The presence of large amounts of ammonia chloride would presumably still further increase the accuracy of the separation.

In the following table there are summarised some results with varying proportions of copper. The separations are made with soda acetate, without any unusual amount of soda chloride being present. For reasons just stated this circumstance places the acetate separation in its most unfavourable light, and is only adopted on account of the greater constancy of the soda cyanide estimation of copper. Many other means of estimating the separated copper are, of course, indifferent to this demerit of the ammonia salts. In case such are allied to the acetate separation, it is well to remember that ammonia salts are better than soda, and Schwarzenberg's precipitation best of all.

TABLE XVIII.

Present.	Recovered.	Percentage.
0.02 grm.	0.0196	97.8
0.03 "	0.0294	98.0
0.05 "	0.0489	97.8
0.10 "	0.0986	98.6
0.20 "	0.1956	97.8

Copper in Steel.

When estimating copper in steel and such iron compounds as contain less than half per cent, it is necessary to weigh off from 5 to 10 grms. of the sample. Such large amounts of iron could not be precipitated as basic acetate without involving a very cumbersome volume of liquid.

The procedure in such cases (and in many others which will suggest themselves) so as to make use of the soda cyanide titration is as follows:—

Dissolve the sample in dilute sulphuric acid, and precipitate as sulphide with sulphuretted hydrogen, or as subsulphide with soda hyposulphite. The latter is perhaps the more convenient for occasional use, and for the

sake of clearness we will describe its processes beyond the point at which they diverge from the usual instructions.

It is very convenient to perform the preliminary operations in a flask. Having filtered the supernatant solution from the precipitated subsulphide, wash two or three times by decantation. The precipitate settles so readily that little more than traces of copper will have passed on to the small paper. Ignite it, replace the residue in the flask, and add a small quantity of nitro-hydrochloric acid—10 to 20 c.c. Heat to boiling, and add separately a few crystals of potassium chlorate. A large excess of hyposulphite should be avoided, else the decomposition at this stage becomes troublesome. When no solid matter remains save a few pieces of sulphur, cool, dilute, neutralise, make alkaline with soda carbonate, and titrate with standard cyanide as previously explained.

It is unnecessary to offer any array of figures in this connection. The separation is one already well established, and that the estimation may be made as proposed has been abundantly proved.

LABORATORY NOTES.

By H. JERVIS.

A CASUAL visit to many laboratories reveals the presence of pieces of apparatus which would be far less cumbersome if its composite pieces had been such as were known to be obtainable, but considered to be too expensive.

A common example of this is seen in the number of makeshifts for tubulated bottles and other articles requiring a hole here or there. This of itself would show that the ability to perforate glass is not largely possessed by the everyday operator.

The process is so serviceable that a few lines explaining the *modus operandi* will be acceptable to many who have never previously tried to do the job, and perhaps helpful to those who have previously left the matter to the "engineering shop," and finally concluded that it needed special apparatus. The operation really belongs to that great class of "tricks" practised by itinerant repairers and scientific amateurs.

Take a hole through a cover-glass as the simplest case. Make a few scratches in the form of an asterisk with the point of a broken three-square file dipped in turpentine. Rest the cover on a wooden block or piece of cork; then turn the sharp point of a similar file backwards and forwards on the mark, or use the point of a broken file fitted in a brace, and the hole is through in a few minutes. In some cases, usually with sheet glass, it is better to bore from each side.

Take now a Winchester. Say we need an aspirator or some form of H_2S apparatus, and want to make a hole $\frac{1}{2}$ inch diameter. Scratch on the asterisk. If only old files are available, use first the point of a three-square to start the hole. Then break the point off and work with new and larger point, and so on until the hole is $\frac{1}{4}$ or $\frac{1}{2}$ inch diameter. A smart tap will break the file as required, and the new edges are very able cutters. When the indentation is thus enlarged, one of the sharp corners of the broken file can be used to groove it towards the centre with almost as much facility as a bradawl could be used on an indent in hard wood. A few turns of the file clears off the ridges and leaves the indent much deeper. If the hole is much cupped and nearly through, a smart tap with a file tang invariably makes a clean hole: there must be no hesitation in giving the tap and no blundering brute force. The hole made, it can be readily widened by working with a tapering file. An ordinary flat file is as good as any other shape, perhaps better—it does not jam so readily. Porcelain can be similarly perforated.

Throughout these operations the tool should be moistened with turpentine, or turpentine and camphor,

and such articles as bottles can be conveniently fixed in position, resting on a partly opened heavy drawer. As an alternative the boring may be done under water in a wash basin or deep sink. In this latter way, too, it is possible to shape sheet glass with a pair of scissors, but one must make haste carefully.

To those whose while it is worth a set of bits made from old files will suggest itself. The Archimedean brace is a good variety for small work. For very small holes the bow brace used largely by watchmakers and crockery riveters is an acquisition which can be readily made.

The breaking of thick glass combustion tubing is really an easy operation. Teachers often break it by heating a nicked portion between coils of wetted paper, and dealers by working on brown paper with string; twine soaked with alcohol is another means. I have found all to be inferior to the following. Make a decided scratch at one portion of the tube, and then complete the circle in a fainter line—not necessarily fainter. An iron rod ($\frac{1}{2}$ or $\frac{3}{4}$ inch) meanwhile pushed up the burner of a muffle will now have become hot; lay it on the deeper scratch until it approaches blackness, and then, if the fracture has not already occurred, touch the heated glass with the tip of a wetted finger. The fracture is in most cases as even as possible. Occasional irregularities are levelled up with the pliers. The effect is greater and the danger of fracture less if the pliers are used from the outer diameter of the tube.

ON THE ESTIMATION OF LIME, ALUMINA, AND IRON IN MINERAL PHOSPHATES.

By M. L. LINDET.

THE attention of agricultural chemists has often been drawn to the part played by alumina and oxide of iron in the retrogradation of superphosphates, and to the difficulties which attend the estimation of these two elements in commercial phosphates. The numerous methods which have been proposed to effect this estimation all require a certain amount of delicate manipulation, and many of them give but uncertain results; they have further been the subject of a critical examination by M. Lasne (*Bull. Soc. Chim.*, pp. 118, 148, 237, 1896), which enables me to dispense with any description of them or to point out their relative disadvantages. The estimation of lime is, as a rule, conducted in the liquors from which the iron and alumina have already been separated; its exactness therefore depends on the processes to which I have just referred.

The most generally used method for the estimation of phosphoric acid consists of precipitating as ammonio-magnesian phosphate, in the presence of a large excess of citrate of ammonia, which keeps the lime, alumina, oxides of iron, manganese, &c., in solution. To then separate these oxides from the filtrates it is necessary to destroy the citric acid, either by evaporation, which never takes place without bumping, and calcination of the residue,—and this is always a long operation,—or by oxidation of this residue with fuming nitric acid, or a mixture of nitrate and chloride of potassium. These oxidations are in general very incomplete, inasmuch as the iron and alumina remain, in spite of everything, in solution, in the presence of ammonia.

It occurred to me that one might, with advantage, and for the purpose of destroying this citric acid, use the beautiful reaction recently described by M. Villiers (*Comptes Rendus*, cxxiv., p. 1349), that is to say, the oxidation of organic matters by nitric acid in the presence of manganese. The reaction, as a matter of fact, can give and has given perfect results.

The operation should be conducted in the following manner:—The ammoniacal liquors, from which the ammonio-magnesian phosphate has been removed, are saturated with nitric acid, and 0.5 grm. of sulphate or nitrate of manganese, and about 50 c.c. of nitric acid for every 20 grms. of citric acid, are then added. This mixture placed in a flask is gently heated, and the attack proceeds during the evaporation; nitric acid is added every time the action decreases; this can be easily seen by removing the flame. When a fresh addition of acid no longer causes any evolution of gas, we may rest assured that there is no longer any nitric acid present, and that it can no longer prevent the precipitation of iron and alumina by ammonia. The precipitate is collected and re-dissolved, and separated by the ordinary methods.

Chloride of vanadium (dichloride of vanadyle, VaOCl_2) may with advantage be substituted for the salts of manganese. Its action is much more energetic, and 0.1 grm. suffices for the rapid oxidation of 20 grms. of citric acid. Hypo-vanadate of ammonium, precipitated at the same time as the iron and alumina, is insoluble under the conditions of the experiment, above all in the presence of ammonia in excess. Instead of trying to separate the iron and the alumina, we can subtract from the weight of the calcined precipitate the weight of oxide of vanadium added; it therefore suffices to make use of a 1 per cent solution of chloride of vanadium, and to take, for precipitating with ammonia, 10 c.c. of the liquid, in presence of a known weight of sesquioxide of iron.

Whether we use salts of manganese or salts of vanadium for the destruction of the citric acid, it is easy, in the liquors from which the iron and alumina have been eliminated, to estimate the lime in the ordinary manner, —*Journ. de Pharm. et de Chim.*, Series 6, vol. vi., No. 6.

THE CALCINATION OF CARBONATED MANGANIFEROUS MINERALS, AND THEIR ASSAY.*

By N. DEVISSE.

BEFORE commenting on the different metallurgical appliances for the calcination of carbonated manganiferous minerals, it is indispensable to point out the thermo-chemical properties, whose interpretation will largely contribute to the elucidation of divers phenomena, and to the determination of the best conditions to allow of the minimum consumption of fuel.

In the first place, the chemical composition of these minerals, when subject to ordinary atmospheric conditions, is in a state of unstable equilibrium, tending towards the formation of pyrolusite, the original pink mineral becoming black. Owing to this conversion, heaps of manganiferous ore have been known to so alter that the percentage of manganese increased from 46 to 52 in a few years.

Diallogite, under the influence of air and rain, behaves in a similar manner, while the rain waters—by dissolving the carbonate of lime and magnesia which are generally present—render the mass porous, enriching it considerably, as in the previous case.

This metamorphosis is further in harmony with the thermo-chemical laws of M. Berthelot, according to which for 1 ton of ore, at 45 per cent contents of Mn, there would be a disengagement of 63.47 calories, equivalent to the heat of combustion of 8.4 kilos. of coal, from which we may conclude that pure diallogite is a combustible material.

This being established, we can now approach the study of the calcination of carbonated manganiferous minerals.

The roasting of these minerals may be carried out

* Abridged from the *Revue Universelle des Mines et de la Metallurgie*, Series 3, vol. xxxix., August, 1897.

either in heaps, kilns, or ovens. The first may be passed over. In new installations the choice of apparatus depends on the quantity of silica present in the mineral; if it is low, kilns are the best to use, having at the same time one or two ovens for the calcination of lumps when their proportion in the ore to be treated is considerable. If the ore contains as much as 8 per cent of silica, it is better to use only ovens.

As soon as the ore attains the temperature necessary for the dissociation of the carbonic acid, this silica unites energetically with the protoxide of manganese, forming a silicate, agglomerating the ore in masses whose volume will frequently entirely upset the operation, the charge descending in the most unexpected manner. It is then necessary to empty the furnace and break up the hard lumps, in which good crystals of the silicate may be often found.

In using the kiln, the ore and the fuel charged at the top absorb the heat of the products of combustion as they ascend, while the air entering below is warmed at the expense of the already calcined material; on arriving at the intermediate zone the air gives up its oxygen, producing, in conjunction with the combustion of the fuel and of the protoxide of manganese, the heat which will be absorbed by the dissociation of the carbonic acid and in the upper zone of decarbonisation.

The calcined ore is drawn out from time to time, at regular intervals, stopping when it appears warm, and the space formed at the furnace mouth is filled with a fresh charge.

Although pure diallogite is a combustible mineral, we must not conclude that the carbonated manganiferous ores, when once alight, will continue roasting of themselves; there is none in nature sufficiently pure to effect this.

Being always in conjunction with the isomorphous carbonates of lime and magnesia, with a certain proportion of silica, it requires for its calcination such a quantity of heat that we must still have recourse to fuel, but in amounts smaller according to the efficiency of the furnace, which must allow of the easy combustion of the protoxide of manganese and the regular descent of the charge.

The combustion of the protoxide of manganese depends on the uniform distribution of a sufficient quantity of air; this is best obtained by means of a damper. The regular descent of the charge depends entirely on the shape and section of the kiln, ovoids, and cylindro-conical furnaces; widening towards the top should be absolutely rejected, the best shape being cylindro-conical in vertical section, slightly widening towards the base. This form allows of the uniform descent of the charge and the parallel ascent of the currents of gas.

The section of the outlet should not be too large, or there will always remain a quantity of ore which cannot be removed.

Among the well-known blast-furnaces used, the best would probably be the Ayresome (Cleveland), and it is to this class that we are indebted for the principle on which the above-mentioned one was constructed.

When the ore to be calcined is siliceous, or for the calcination of large lumps, we have already said that the kiln is unsuitable; in these cases it is best to make use of large parallel ovens, about 7 metres long by 1·5 metres wide, and about 8 metres high. The floor which carries the ore is formed of rows of bricks, with spaces in between of about 0·15 metre in width, forming an arch 0·35 metre high in front of each oven door. The oven being empty, we begin by charging it with the unroasted ore from the previous operation, to about 0·25 metre thickness, putting the largest lumps over the spaces between the bricks; we then spread the first layer of fuel, and alternate layers of fuel and raw ore up to 0·4 metre high. When the charge reaches the height of the door it is bricked up, and the fire started with wood under each arch. The charging continues day by day, taking care to gradually diminish

the quantity of fuel, especially for the last two layers, when finer ore must be used. As soon as the first charge becomes of a dull red heat, the fire below is allowed to go out, and the combustion proceeds from layer to layer. After four or five days the fire reaches the upper layers; when cool the door at the bottom is opened, and the mineral—which is in what is called the "liquid" state—runs out with ease. The operation lasts eight or nine days from beginning to end, and gives a return of about 400 tons per month.

The problem of calcining pulverulent minerals has not yet been satisfactorily solved; it cannot be done in a kiln or an oven as just described. It has been done in a "Pelatan" furnace, but at the expense of 12 to 13 per cent of coke.

The Assay of Ores.

Without reviewing the known methods of estimating manganese, we will simply point out a few facts deduced from the beautiful experiments made by Gorgeu (*Ann. de Phys. et de Chim.*, "On Manganous Acid," Series 3, vol. lxvi., p. 153), the importance of which does not seem to have been sufficiently appreciated.

When we precipitate manganese in the presence of an oxidising agent, such as chlorine, bromine, &c., this metal tends to separate in the form of manganate, according to the formula $5\text{MnO}_2, \text{MO}$, MO being either protoxide of manganese, oxide of zinc, lime, &c. For example, when we add permanganate to chloride of manganese all the permanganate is decomposed; at the same time the liquid becomes acid, and a brown precipitate takes place. If we neutralise the acid set at liberty, as it is produced, we notice, after having added one equivalent of permanganate of potash to four of chloride of manganese, that three-quarters of the hydrochloric acid have been set at liberty, and that there is no longer any manganese remaining in solution:—



The presence of salts of lime, zinc, &c., thus casts suspicion on gravimetric estimations, while on the contrary it is necessary to the accuracy of volumetric methods, in which the object is to measure the quantity of oxygen absorbed by the manganese in its precipitation in the state of binoxide, or, rather of manganous acid. We strongly recommend the use of volumetric methods, which are of extreme accuracy whenever we add to the solutions a sufficient quantity of a soluble salt of zinc or lime; we even advise keeping a certain quantity of these bases in suspension at the moment of precipitation.

Under these conditions all the manganese will be precipitated in the state of manganous acid, in the manganites of zinc or lime which are formed; the manganite of manganese is not formed except when this metal is alone, as, for example, when we submit precipitated carbonate of manganese to the action of chlorine in excess.

Finally, in Volhard's method, the most elegant and rapid of all the volumetric methods, it is not necessary to transform the chlorides into sulphates, if we make it a rule to add precipitated oxide of zinc to the dilute neutral liquid to which the permanganate of potash is to be added. In fact, the hydrochloric acid set at liberty in the reaction—



is in this manner neutralised immediately on its formation.

Jubilee Medal.—The Queen has been graciously pleased to bestow upon Mr. Walter Hills, President of the Pharmaceutical Society of Great Britain, a mark of Royal favour in connection with the sixtieth anniversary of Her Majesty's reign, by presenting him with a medal to be worn as a decoration commemorative of that event.

ON THE COMPOSITION OF CERTAIN
CANADIAN VIRGIN SOILS.*By FRANK T. SHUTT, M.A. F.I.C., F.C.S.,
Chemist, Dominion Experimental Farms.

(Continued from p. 206).

North-West Territories and Manitoba.

THE prairie soils of the North-West Territories and Manitoba are justly noted for their productiveness. They contain, as a rule, large percentages of all the essential constituents, and are characterised by percentages of humus and nitrogen far above the average. The prevailing surface soil, speaking generally, is a black or greyish black loam in which the vegetable matter is well decomposed and thoroughly incorporated with the inorganic compounds of the soil. It varies in depth from a few inches to one, two, or even more feet, and over large areas is underlain with a heavy clay subsoil.

Occasionally we have had sent to us soils from certain districts in the North-West Territories, in which it is stated that poor yields are obtained. On examination, these soils have been found to possess plant food in adequate quantities for crop requirements. Further, they have usually been found to be free from alkali. Investigation has shown that the trouble was, not in the lack of plant food, but rather in the climate; a scanty rainfall being really the cause of the poverty of growth. In districts subject to drought irrigation, if feasible, would render such soils most fertile. An illustration of this is afforded by the late irrigation trials at Calgary, which have proved so successful from an agricultural point of view. In this connection we have to add that unfortunately no means for extensive irrigation appear practicable for several of the districts here referred to in the North-West Territories.

The presence of "alkali" in the soil in patches over certain areas in Manitoba and the North-West Territories is intimately connected with the question of rainfall. An alkali area may be restricted to a few square feet, or it may cover some acres. Patches of alkali soil occur surrounded by land of great productiveness.

The formation and retention of alkali are dependent upon the amount of water the soil receives and the facility for subsoil drainage. We need not now discuss the occurrence of alkali nor its nature, but it is valuable to note that, though the amounts of alkali found in samples submitted to us are often so great as to render the growth of wheat impossible, we have invariably found such soils to be rich in mineral and organic constituents. This shows that the soil proper is capable of acting as a fertile one, provided the alkali were got rid of by drainage, irrigation, or treatment with gypsum.

In Table III. we have given analytical data of seven surface soils from the North-West Territories. Though there is a greater uniformity in the texture and composition of soils upon the prairies than among soils of the Eastern provinces, no claim is made that the vast extent of the Territories is represented by these samples—they are altogether too few in number. They may serve, however, to indicate the general character of the soils over certain large areas.

Without discussing these soils in detail, attention may be called to their high nitrogen content and the large amounts of organic matter that are almost invariably present. These soils also contain above the average amount of potash. Our results do not show them to be noted for phosphoric acid, though they possess quantities quite equal to those in many very fertile soils. The great depth of the surface soil over large areas accentuates our deductions respecting the vast stores of plant food laid up in the plains for future crops. We are of the belief that

where poor crops only are procurable the climatic conditions are rather at fault than that there is a lack of plant food. Even in soils containing injurious amounts of alkali we have found, as already pointed out, an abundance of fertilising ingredients; drainage, if there is an adequate rainfall, frequently being all that is necessary to bring them into a state of productiveness.

Soil No. 37 represents the unfertilised and uncropped prairie soil of the Red River Valley, Manitoba. It was taken from Section 31, Township 4, Range 1, West. The uniformity in the character of the soil over a very large area in Manitoba makes the data here presented of more than ordinary importance.

The surface soil, which is fairly uniform throughout its depth, averages a little over 2 feet in thickness and merges gradually into the subsoil, which is blue clay. The latter, as tested by boring for water at this spot, extends at least to a depth of 250 feet.

The soil is deep black loam, of a fine and peculiarly characteristic granular order. It reduces easily between the fingers in the air-dried condition to a greyish brown powder. Though there is present a considerable amount of undecomposed root-fibre, the soil proper exhibits a remarkable homogeneity, indicating a process of physical refining in its formation and a uniformity in the chemical composition. The very large amount of organic matter present is undoubtedly most intimately incorporated with the clay and sand which constitutes the basis of the soil.

Though containing a large amount of clay, laboratory experiments show that this soil does not readily "puddle" on moistening, nor on subsequent drying does it form into a hard mass, but readily granulates on slight pressure.

The large amount of organic matter present has already been remarked; it exceeds 25 per cent of the water-free soil. The nitrogen is found to be practically 1 per cent, which would show that there is contained in an acre of soil to the depth of 1 foot more than 30,000 pounds of this element. Since ordinary fertile soils to a like depth contain from 3500 to 10,000 lbs. of nitrogen per acre, the vast reserve of this valuable constituent in this prairie soil is apparent.

This soil is also very rich in potash, containing an amount far in excess of that ordinarily met with in fertile soils. But two other virgin soils examined by us approach its potash content, 1.03 per cent.

Of phosphoric acid it contains 0.29 per cent. This also is above the average, most of our good soils showing between 0.15 per cent and 0.25 per cent phosphoric acid.

We may safely conclude that there is here ample scientific proof of the well-nigh inexhaustible stores of plant food, and that this prairie land, as regards the elements of fertility, ranks with the richest of known soils.

Concerning the prairie soil of the Red River Valley, Dr. Geo. M. Dawson, Director of the Geological Survey of Canada, wrote some years ago as follows:—

"Of the alluvial prairie of the Red River much has already been said, and the uniform fertility of its soil cannot be exaggerated. The surface, for a depth of two to four feet, is a dark mould, composed of the same material as the subsoil, but mingled with much vegetable matter. Its dark colour is no doubt due in part to the gradual accumulation of the charred grasses left by the prairie fires. The soil may be said to be ready for the plough, and in turning the tough thick prairie sod, the first year a crop of potatoes may be put in, though it is not efficiently broken up till it has been subjected to a winter's frost. When the sod has rotted, the soil appears as a light friable mould, easily worked and most favourable for agriculture. The marly alluvium underlying the vegetable mould would, in most countries, be considered a soil of the best quality, and the fertility of the ground may, therefore, be considered as practically inexhaustible.

"The area of this lowest prairie has been approximately stated as 6900 square miles, but the whole is not at present suitable for agriculture. Small swamps are scattered pretty uniformly over its surface. The greater

* Read before the British Association (Section B), Toronto Meeting, 1897.

TABLE III.—Analyses of Soils (Water-free), North-West Territories and Manitoba.

No.	Locality.	Surface or subsoil.	Character of soil.	Potash.	Phosphoric acid.	Nitrogen.	Lime.	Loss on ignition (organic and volatile matter)
30.	Yorkton, N.W.T. ..	Surface.. ..	Black sandy loam ..	0.49	0.21	0.504	0.06	14.01
31.	" " ..	Subsoil.. ..	" " " " ..	0.42	0.09	0.130	0.75	8.18
32.	Saltcoats, " ..	Surface.. ..	" " " " ..	0.34	0.21	0.571	2.90	13.54
33.	Moosomin, " ..	" " " " ..	Black loam.. ..	0.36	0.11	0.479	0.95	11.79
34.	Calgary, " ..	" " " " ..	" " " " ..	0.44	0.17	0.447	0.92	12.23
35.	Tilley Township, N.W.T. ..	" " " " ..	" " " " ..	0.27	0.18	0.398	0.37	11.13
36.	Vermillion Hills, N.W.T. ..	" " " " ..	" " " " ..	0.17	0.17	0.354	0.50	10.43
37.	Red River Valley, Manitoba ..	" " " " ..	" " " " ..	1.03	0.29	1.005	1.89	26.29

TABLE IV.—Analyses of Soils (Water-free), Ontario.

38.	Sinclair Township, Muskoka ..	Surface.. ..	Sandy loam . . .	0.11	0.27	0.186	0.12	8.74
39.	Chaffey Township, Muskoka ..	" " " " ..	" " " " ..	0.08	0.12	0.139	0.40	6.79
40.	Chaffey Township, Muskoka ..	Subsoil.. ..	Sand	0.08	0.18	0.074	0.20	3.53
41.	Franklin Township, Muskoka ..	Surface.. ..	Light grey loam .	0.61	0.18	0.103	0.76	6.31
42.	Franklin Township, Muskoka ..	Subsoil.. ..	" " " " ..	0.02	0.08	trace	0.66	3.70
43.	Perry Township, Muskoka ..	Surface.. ..	Sandy loam . . .	0.04	0.18	0.296	0.08	9.40
44.	Perry Township, Muskoka ..	Subsoil.. ..	" " " " ..	0.06	0.18	0.119	0.13	5.10
45.	Brunel Township, Muskoka ..	Surface.. ..	Clay loam	0.46	0.17	0.084	1.28	2.94
46.	Brunel Township, Muskoka ..	Subsoil.. ..	" " " " ..	0.29	0.09	0.064	1.07	2.39

TABLE V.—Analyses of Soils (Water-free), Quebec.

47.	Arthabaska, Quebec	Surface.. ..	Sandy loam . . .	0.16	0.17	0.296	0.35	8.68
48.	" " "	Subsoil.. ..	" " " " ..	0.17	0.18	0.184	0.29	5.46
49.	St. Adelaide de Pabos	Surface.. ..	Red sandy loam..	0.44	0.07	0.215	0.16	7.85
50.	Soulanges, Gaspé..	" " " " ..	Grey sandy loam ..	0.39	0.33	0.198	0.47	7.76
51.	" " "	Subsoil.. ..	" " " " ..	0.47	0.30	0.049	0.73	3.67
52.	Lievre River, " ..	Surface.. ..	Clay loam	0.11	0.19	0.179	1.23	5.77
53.	" " "	Subsoil.. ..	" " " " ..	0.10	0.19	0.171	1.17	5.62
54.	Joilette, " ..	Surface.. ..	Black clay loam..	0.40	0.28	0.218	0.82	8.06
55.	" " "	Subsoil.. ..	" " " " ..	0.44	0.29	0.030	1.05	2.09
56.	Bonaventure, " ..	Surface.. ..	Reddish yellow clay loam	1.17	0.19	0.249	0.10	12.37

part of these swamps are, however, so situated as to be easily drained, either into the Red River or some of its tributaries, which are usually depressed 30 or 40 feet below the level of the surface.

"As a measure of the possible agricultural capacity of this great valley, take one-half of the entire area, or 3,400 square miles, equalling 2,176,000 acres, and for simplicity of calculation, let it be supposed to be sown entirely in wheat; then, at the rate of 17 bushels per acre, which, according to Prof. Thomas, is the average yield for Minnesota, the crop of the Red River Valley would amount to 40,992,000 bushels."

Ontario.

The review of soils in this province will be restricted to certain surface and subsoil samples collected in the district of Muskoka (Table IV.)—a district lying somewhat over 100 miles north of Toronto, and considered, for the most part, more picturesque than agricultural; it is rocky and abounding in lakes, well timbered—save where destructive fires have swept through,—with stretches of fairly good, though, as a rule, light soils along the river valleys and

on the tower levels. Our data respecting virgin soils in other parts of the province are too fragmentary to warrant their insertion in this paper.

Soil No. 38.—From Sinclair Township. A shallow very loose sandy soil; the subsoil of hard pan is found at a depth of from 6 to 12 inches. Though moderately rich in phosphoric acid, nitrogen, and humus, it is below the average in potash and lime.

Soils Nos. 39 and 40.—Surface and subsoil from Township of Chaffey. A shallow sandy loam running into a subsoil of sand. Hard pan exists at a depth of 15 inches. The surface soil is deficient in potash, but otherwise of medium quality as regards plant food.

Soils Nos. 41 and 42.—From Franklin Township. The surface soil is a light grey clay loam, high in potash, fair in phosphoric acid, and low in nitrogen; lime is present in an amount that might be considered large for Muskoka soils.

Soils Nos. 43 and 44.—Perry Township, Parry Sound district. Soil and subsoil. The country is described as level or gently sloping, with no rocky bluffs, and well timbered with excellent hardwood.

Both samples are light and sandy in character, and exceedingly low in potash and lime. Regarding the surface soil, we may say that the percentage of phosphoric acid is fair, and that in nitrogen it is above the average, compared with soils of this character in this district.

Soils Nos. 45 and 46.—Surface and Subsoil from Brunel Township. The surface soil is a clay loam of a light grey colour, from 8 to 12 inches in depth. It is a fairly strong and retentive soil, and in this respect differs from the preceding members of this series. The features in its favour are the comparatively high percentages of potash and lime. In nitrogen and humus, however, the soil is poor.

It is thus seen that the soils of this northern part of Ontario are characterised by a preponderance of sand, the larger number being such as would be classed as light or very light loams. They are loose in texture and very apt to dry out in season of drought. Though scarcely heavy enough for wheat, they grow good crops of oats and potatoes. Being responsive to manures, large yields of root and fodder crops can, under good system of culture, readily be obtained in favourable seasons. The district is better adapted for grazing and dairying than for the growth of cereals.

Quebec.

Table V. presents the data obtained from the examination of ten soils from the province of Quebec. They, as the preceding samples, have been selected as typical average soils; not, on the one hand, representing the richest, nor, on the other, the poorest, lands.

Soil No. 47.—Surface soil from Arthabaska county. A sandy loam of fair quality; nitrogen and organic matter are present in quantities somewhat above the average, but the soil ranks rather low as regards mineral constituents.

Soil No. 48.—Subsoil to the above, and very similar in its proportion of potash and phosphoric acid. For a subsoil it may be considered high in nitrogen.

Soil No. 49.—A surface soil from Gaspé. It is a red sandy loam, containing fair quantities of potash and nitrogen, but low in phosphoric acid and lime.

Soil No. 50.—A dark grey sandy loam from Soulanges county. A light, warm, responsive soil. In all the elements of plant food it may be placed with soils of good average fertility.

Soil No. 51.—Subsoil to the above, in which the mineral elements are present in fair amounts.

Soil No. 52.—A heavy clay loam from the valley of the Lièvre River, Ottawa county. A strong retentive soil. With drainage it should be well adapted to the growth of cereals. Though low in potash for a clay soil, it may be regarded as of average fertility. Drainage and the application of lime have vastly improved its productiveness.

Soil No. 53.—Subsoil to above, and very similar to it, both chemically and physically.

Soil No. 54.—A clay loam from Joliette county; greyish black in colour, compact, and cohesive. Both in mineral constituents and nitrogen this soil is above the average. An application of 20 bushels of lime per acre, however, resulted in almost doubling the yield.

Soil No. 55.—Subsoil to No. 54. Stiff clay, grey to reddish brown.

Soil No. 56.—A surface soil from the county of Bonaventure. A reddish yellow loam, containing a slight preponderance of sand. The large amount of iron present masks the presence of the organic matter, of which there is a notably high percentage. Not unfrequently—indeed, one may say usually—a rough estimate of the organic matter—and, incidentally, nitrogen—present can be made from the colour of the air-dried soil. In soils, however, such as the one under discussion, containing high percentages of iron, the colour can no longer be used as a criterion of the soils richness in these constituents.

Much variation, as might be expected, in character and composition is to be observed among these soils. Though some possess but small amounts of certain constituents,

indicating inadequate quantities for the best returns, yet none fall below the limits of fertility previously discussed, and many are seen to compare most favourably with soils of recognised productiveness.

(To be continued).

EARLY AMERICAN CHEMICAL SOCIETIES.*

By Prof. H. CARRINGTON BOLTON.

THREE chemical societies were organised in the United States before the close of the first quarter of this century; one as early as 1792, the second in 1811, and the third in 1821. These societies were short-lived, local in jurisdiction, and without much influence on the progress of the science; but it is interesting to note that professional, teaching, and amateur chemists in America formed associations for mutual improvement, and for the advancement of their calling, long before their European brethren. The Chemical Society of London, the oldest in Europe, was founded in 1841, forty-nine years after the first American society; that of Paris dates from 1858, and that of Germany from 1868. American chemists were not impelled to form independent societies, owing to a lack of organisations for men of science, but they early felt the advantages of a specialised association. The society of 1792, and that of 1811, were both founded in a city honoured by the presence of the venerable and dignified American Philosophical Society, established by Benjamin Franklin in 1743.

The existence of these societies has long been known, but only through casual references to them by writers on the beginnings of science in the United States; Prof. Benj. Silliman, in his essay on "American Contributions to Chemistry," read at the centennial celebration of the discovery of oxygen, held at Northumberland in 1874, alludes to them incidentally; and Dr. Brown Goode, in his historical addresses to the Biological Society of Washington, barely mentions them.

The publications too, of the earlier societies, are very little known, being rarely found in the best libraries.

Under these circumstances it has seemed not altogether useless to summarise what information concerning these societies I have been able to gather, and to offer it as a contribution to the history of chemistry in the United States.

The three societies are:—

I. The Chemical Society of Philadelphia, founded in 1792.

II. The Columbian Chemical Society of Philadelphia, founded in 1811.

III. The Delaware Chemical and Geological Society, founded in 1821.

I. The Chemical Society of Philadelphia.

The Chemical Society of Philadelphia was undoubtedly the earliest organised body of chemists in either hemisphere, having been "instituted" in 1792. The society does not seem to have published records of its meetings, nor of the papers presented thereat, and, since at that early day the primitive local newspapers paid little attention to items of scientific interest, information concerning it is not readily obtained. I find, however, that it was flourishing in 1801-2, when it had the following officers:—

President—Dr. James Woodhouse.

Vice-Presidents—Felix Pascalis and John Redman.

Librarian—William S. Jacobs.

Curators—William Brown and John S. Dorsey.

Treasurer—John Y. Bryant.

Secretary—Thomas Brown.

The society held stated meetings each week.

The President of the Society, Dr. James Woodhouse

* Read before the Washington Chemical Society, April 8, 1897. From the *Journal of the American Chemical Society*, August, 1897.

(1770—1809), was at the time professor of chemistry in the medical department of the University of Pennsylvania, of which he was a graduate.

This chair had been held by Dr. James Hutchinson, and on his death, in 1793, Dr. Joseph Priestley, who arrived from England a few months later, was invited to succeed him, but he declined, preferring the quiet life of Northumberland, and Dr. Woodhouse was chosen instead. Dr. Woodhouse contributed several medical papers to the New York Medical Repository, and to Cox's Medical Museum; he also edited Chaptal's "Elements of Chemistry" (fourth edition, 1807, 2 vols.), and Parkinson's "Chemical Pocket-Book" (1802). He is said to have been the first to prove by comparative experiments the superiority of anthracite coal from Pennsylvania over bituminous coal from Virginia for intensity and regularity of heating power (Silliman).

The first vice-president, Felix Pascalis Ouvrière (1750-1840), had an interesting career. He was born in France, where he received his medical education; he emigrated to Santo Domingo, and while practising medicine there acquired an extensive knowledge of botany and other branches of natural history. In 1793 a revolt among the negroes compelled Pascalis to flee, and he took refuge in the United States, first at Philadelphia, and later at New York, where he resided for more than thirty years. He was the founder of the Linnean Society of New York, and the author of several medical papers and reports.

The second vice-president, Dr. John Redman, (1722-1808), was a native of Philadelphia, and educated in European medical schools and hospitals. In 1786 he was made president of the Philadelphia College of Physicians. He was regarded as one of the foremost practitioners of medicine of Philadelphia, but his methods now appear super-heroic.

Dr. John Syng Dorsey (1783-1818), one of the curators, was professor of surgery and afterwards of materia medica in the University of Pennsylvania. He had a high reputation as a surgeon, but his qualifications for membership in a chemical society seem to have been based chiefly on the fact that in his youth he had attended the chemical lectures of Sir Humphry Davy (1803).

I have not found the roll of members of this early society, but it appears that Priestley, Hare, and Seybert were active in it. The ambition of the members is shown by the circumstance that in 1802 there was a standing committee prepared to "annalize every mineral production" brought before them, and to give "an accurate account of each specimen free of expense."

The meeting held October 24, 1801, was made memorable by the appointment of a committee for the "discovery of means by which a greater concentration of heat might be obtained for chemical purposes." On this committee was placed among others Robert Hare, then only twenty years of age; but so soon as December 10th of the same year he reported to the society, on behalf of the committee, his invention of the "hydrostatic" (oxy-hydrogen) blowpipe. I need not here eulogise this important and useful invention, which yielded such a fruitful harvest of discoveries. This alone justified the existence of the first of chemical societies. In the following year the society caused Dr. Hare's account of this blowpipe to be printed in a pamphlet of thirty-four pages, 12mo., with one plate. This now rare booklet bears the title "Memoir on the Supply and Application of the Blowpipe, containing an account of a new method of supplying the blowpipe either with common air or oxygen gas; and also of the effects of the intense heat produced by the combustion of the hydrogen and oxygen gases. Illustrated by engravings. Published by order of the Chemical Society of Philadelphia, to whom it was presented by Robert Hare, jun., Corresponding Member of the Society. Philadelphia. Printed for the Chemical Society by H. Maxwell, Columbia House, 1802."

* A copy of this is found in the Army Medical Library, Washington, D.C.

Robert Hare's subsequent career as professor of chemistry in the medical school of the University of Pennsylvania from 1818 to 1847, is well known and accessible to all enquirers.

How much longer this association of chemists continued to meet, I have not ascertained. But the work of this society was evidently remembered by those who, ten years later, founded a new one, inasmuch as they designated it by the prefix "Columbian" to avoid confusion.

(To be continued).

THE PRODUCTION OF HALOIDS FROM PURE MATERIALS.*

THIS Report consists mainly of an abstract of portions of a paper lately published by the Secretary in the *Journal of the Chemical Society*.

It is well known that many chemical changes depend upon the presence of water among the acting substances, and some chemists have been tempted to suspect that possibly chemical change may not occur at all in the absence of moisture. On the other hand, there remain a very substantial number of changes which have not hitherto been "stopped" by the careful withdrawal of water from the sphere of action. Among these are the forming of ozone from oxygen, and the combining of certain metals (for example, mercury) with chlorine.

For some time past members of the Committee have been occupied in re-examining some of these latter phenomena. Great care was taken in preparing the various materials required; and novel and stringent methods of drying them were employed. Advantage has been taken of the opportunities which occurred during the progress of the work to re-examine the influence of sunlight, and of the silent electric discharge on highly purified chlorine.

The following is a summary of the results obtained:—

1. Carefully purified specimens of mercury, made by three distinct methods, were found to combine rapidly and completely with carefully dried chlorine.
2. Carefully purified mercury was also found to combine rapidly and completely with well-tried bromine and iodine.
3. Chlorine prepared by the electrolysis of silver chloride and dried by a brief exposure to phosphoric anhydride is not condensed when submitted to the action of the silent electric discharge.
4. Chlorine from the same source (see 3) becomes more sensitive than before to the action of sunlight, after the addition of a trace of damp air.
5. Lead glass, which is readily corroded when heated in damp chlorine, is unaffected by the same gas after it has been well dried.

As bearing on the general question, it may be mentioned that it was shown in the original memoir (*loc. cit.*) that well-dried ozone undergoes spontaneous decomposition far more rapidly than the damp gas. That is to say, the reaction $2O_3 = 3O_2$ is retarded, and not facilitated, by the presence of water. It remains to be seen if other and analogous reactions, some of which will shortly be investigated, are effected in a similar manner.

Contribution to the Biological History of the Phosphates.—L. Joly.—The solution of ammonium molybdate in dilute nitric acid so often employed in analytical chemistry for the detection of phosphoric acid serves us to detect its presence in animal tissues.—*Comptes Rendus*, cxv., No. 15.

* The substance of a Report of a Committee consisting of Professors Armstrong and Dunstan, and Messrs. J. D. Cundall, C. H. Bothamley, and W. A. Shenstone (Secretary). Read before the British Association (Section B), Toronto Meeting, 1897.

NOTICES OF BOOKS.

The True Chemistry. ("Chimie Vraie"). The Rigorous Application of Two General Laws of Chemical Action. By E. J. MAUMENÉ, Dr. es Sc. Pp. 205. Paris: P. Vicq-Dunod and Co. 1897.

For thirty-seven years M. Maumené has been working on his theory, and claims to have given thousands of proofs of the accuracy of the two laws discovered by him, viz., the "Law of Contact" and the "Law of Mixture." In a previous work, "A Treatise on the General Theory of Chemical Action" (1880), the author admits that some of his calculations were only approximate, and that this might have prejudiced some persons against them; but in this last volume, just published, every calculation is exact, simple, and uniform, and can be followed by any one. These two laws, he claims, will greatly simplify the theory of chemistry, by superseding it, through their simplicity. Basicity, affinity, dissociation—all can be shown, and worked out in plain figures, by one or the other of M. Maumené's two fundamental laws, which make "The True Chemistry."

The essential aim of chemistry is to know exactly what takes place in any change, so that under given circumstances the result of any other reaction can be foretold; this we can already do to a certain extent, but there is generally *something* which does not fit; it may be called the exception which proves the rule, but it may also be said that it is the exception which proves the rule to be wrong. If by calculation all the unknown reactions can be predicted, then it must be admitted that there is much to say in favour of M. Maumené's theory. It must, however, be left for the chemical world at large to judge; true philosophers will always accept new ideas, however startling, when once they are proved beyond any doubt to be correct.

This volume gives ample evidence of an enormous amount of trouble and thought, spread over many years, and the author deserves the thanks of all chemists for the ideas he has given them; it also stands before many other works hailing from across the channel,—it has an excellent index.

The Induction Coil in Practical Work, including Röntgen X Rays. By LEWIS WRIGHT. London: Macmillan and Co.

In view of the considerable interest that has recently been directed to electrical discharge in high vacua by Dr. Röntgen's discovery involving the use of powerful and costly induction coils, the book before us supplies a want, and is likely to be greatly appreciated. The author in his preface explains that his object has been to give practical help to the efficient and safe use of an induction coil; and this idea seems to have been faithfully adhered to throughout the book.

In the first chapter, of twenty-two pages, the simple principles of electrical induction are lightly touched upon,—the woodcuts, by the way, strike us as old acquaintances,—but the matter is fresh and quite up to date.

In Chapter II., after briefly describing the early Du Bois-Reymond apparatus, the modern coil is thoroughly explained, diagrams and drawings being given of the various parts, not as a guide to amateur coil-makers, but simply to give the general principle upon which the modern coil is built.

In Chapter III. the instructions for the "care of a coil" and the use of primary batteries are well worth noting by any who are using this class of apparatus for the first time, for a little neglect will easily ruin a most expensive coil, and by simple attention to the details noted both coil and battery will be kept in good order.

A short chapter is devoted to "Miscellaneous Experiments," probably to whet the appetite of the beginner,

and then three chapters are given to the electrical discharge in rarefied gases; brief accounts are given of De la Rive's experiments, Plücker's researches on stratified discharges, and others.

Chapter VII., on "The Discharge in High Vacua," is almost exclusively devoted to the researches of Sir W. Crookes, whose papers on Molecular Physics and Radiant Matter are extensively quoted and illustrated.

The remaining portion of the book is occupied by a discussion of Professor Röntgen's discoveries and their developments. Lenard's early work on the cathode rays is noted, and then the now old story of the X rays is retold. Some good practical advice is given as to the use of the Crookes tube in practice, which will repay the attention of any one using this expensive and easily damaged apparatus. It is easy to see that the author is writing from practical experience, and his instructions will be found of great value.

There is a very "made in England" style about the book, and the author shows a great—and one is inclined to think almost too great—contempt for Continental apparatus.

A footnote at the end of the book refers to a new form of contact-breaker recently described, in which the chief novelty lies in the fact that the swinging hammer of the break has to swing some distance before the contact is broken, thus making a very sudden break, causing less sparking and more inductive effect. It does not seem to be generally recognised that this principle is embraced in the very simple and primitive form generally used on Continental coils, where the mass of iron forming the hammer is fixed on the end of a somewhat long flexible spring, the platinum contact being placed some inch or so lower than the iron head.

The book is well printed and plenty of illustrations are given, making it both interesting and instructive.

Report of the Council of the Institute of Mines and Forests on the Gold and Forest Industries of British Guiana for the Year ending June 30, 1897. Demerara: W. H. Hinde, Echo Office, 1897.

It is satisfactory to note that the gold industry of British Guiana shows signs of improvement, the output for the year ending June 30, 1897, being 128,333 ounces, as against 119,422 ounces in the previous year. It, however, still falls short of the outputs for 1892-3, 1893-4, and 1894-5, which were respectively 138,279 ounces, 137,822 ounces, and 128,760 ounces.

The past year has been marked by the first returns of any importance from quartz crushing, the Barima mine having produced over 7000 ounces.

Of the five districts into which the gold-bearing territory is divided, the most encouraging increase of all is to be found in the Potaro District; it has shown a steady increase since 1892, and now heads the list with 30,434 ounces, and it is hoped that with the extra facilities afforded by the Demerara-Essequibo Railway and the Government Road that the next twelve months will see a still further increase in the prosperity of this important district.

The number of labourers employed in the gold mining industry throughout the colony on June 30, 1897, was registered as 17,050.

The Governor has placed the services of Prof. Harrison, the Government analyst, at the disposal of the Institute for the examination of minerals free of cost, on condition that the samples are of public interest and that the results are published for general information.

The exports of forest products, including timber, shingles, charcoal, &c., also show a most encouraging increase over last year, especially viewed with regard to the prevailing depression in the staple product—sugar. The export of timber alone has almost doubled itself,

being for the year ending June 30, 1897, 404,234 cubic feet, valued at over 148,000 dollars.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxv., No. 15, October 11, 1897.

Further Experiments on the Liquefaction of Fluorine.—H. Moissan and J. Dewar.—Already inserted.

Direct Transformation of Heat into Electric Energy.—Marcus Deprez.

Spectra of the Coloured Components of Double Stars.—Sir William Huggins.

Spectra of the Principal Stars of the Trapezium of the Nebula of Orion.—Sir William Huggins.

New Mixed Platinous Salt.—M. Véles.—The limited action of the hydrochloric, hydrobromic, or hydriodic acids upon potassium platinonitrate, $\text{Pt}(\text{NO}_2)_4\text{K}_2$, gives rise to the elimination of half the nitrous acid contained in this salt, and to the formation of mixed chloro-, bromo-, or iodo-nitro-salts very stable in aqueous solution; the platino-dichloro-nitrite, $\text{PtI}_2(\text{NO}_2)_2\text{K}_2$; lastly, the platino-diiodo-nitrite already obtained by Nilson. Oxalic acid gives rise to an analogous reaction with the sole difference that, by reason of its basicity, a single molecule of acid in place of two intervenes in the reaction. The properties of potassium platino-oxalonitrite are capable of being utilised in the separation of the platinum metals.

Procedure of Separating and Distilling Bromine from a Mixture of Alkaline Chloride and Bromide.—H. Baubigny and P. Rivals.

Reversible Transformation of Styrolene-meta-styrolene under the Influence of Heat.—G. Lemoine.—The reversible transformation of styrolene into meta-styrolene under the influence of heat recalls by its general course that of phosphorus, cyanogen, and cyanic acid; it tends progressively towards a limit expressed by a tension of the vapour of styrolene.

On Two Coloured Reactions of Pyruvic Acid.—L. Simon.—Pyruvic acid, with the addition of potassa and then of sodium nitroprusside, yields a beautiful and intense violet-red colouration. This is a reaction which appears to belong to all the fatty amines; it has been verified for the three methylamines, for mono- and diethylamine, amylamine, and benzylamine; with the last-mentioned base the colouration is that of lye of wine.

Action of Nitric Acid upon Potassium Cobaltcyanide.—E. Fleurent.—A note which the author merely gives to "take date."

Bulletin de la Société de Pharmacie de Bordeaux.
September, 1897.

Colorimetric Estimation of Manganese.—M. Lemaire.—Manganese is found in small quantities in a large number of plants, and many botanists consider it necessary to their proper nourishment. The ordinary gravimetric and volumetric methods of analysis are not suitable for estimating these small quantities; but, on the other hand, the colorimetric method is very convenient. It depends on the reaction pointed out by Hoppe-Seyler: If, to a substance free from chlorine, and containing manganese, we add binoxide of lead and nitric acid, we obtain, on heating to boiling, a violet colouration due to the formation of permanganic acid. This reaction is very sensitive, and will detect $1/2,000,000$ th part of man-

ganese. The leaves of a sample of wild chicory, dried at 100° , gave 19.20 per cent of ash and 0.0004 per cent of manganese. The roots of another sample contained 0.0002 per cent of this element.

The Agricultural Journal of the Cape of Good Hope,
Vol. xi., No. 5.

Rinderpest Conference.—At the conference held on August 19th on this important subject, the several methods which have been employed for immunising cattle were discussed. It was at first expected that Dr. Koch's method of inoculating the healthy animals with the bile of a diseased one would confer immunisation on them, but there has since been a considerable difference of opinion as to its value. Unfortunately we are not in possession of a simple practical test which will enable us to distinguish between a safe bile, permanent in its effects, and biles which are either too strong or too weak. Even with biles which were to all appearances of standard quality, very varying results were obtained; and, further, it was found that the disease itself could be communicated in this manner. It is this uncertainty which is the most disappointing part of Koch's bile inoculation. Immunity has lasted as long as three or four months, but then the effect seems to wear off. As bile did not give the modified form of disease, it was found to be necessary to inoculate afterwards with virulent blood, and the present experience seems to be that Edington's method of inoculating a second time with virulent blood gives very good results.

Revue de Chimie Analytique.
Vol. v., No. 18.

Calibration of Graduated Glass Vessels.—M. Vandevyver.—The author thinks that the method recommended by M. Démichel, for correcting the error due to surface tension when calibrating glass vessels, is insufficient, as it assumes that the surface tension is constant and invariable, whereas such is not the case. For example, in gradually filling a glass with water, one-quarter at a time, the surface tension varies as follows: $\frac{1}{4}$ full 6.35, $\frac{2}{4}$ full 5.22, $\frac{3}{4}$ full 5.09, full but not overflowing 4.89, full and overflowing 7.32. The author considers the following the best method to adopt for getting accurate results:—The vessel must first be cleaned with the greatest care, by washing it in acetic acid, caustic potash, then ether, alcohol, and finally rinsing several times with distilled water. It is then dried by inspiration, taking care to keep out dust by lightly plugging with cotton-wool, through which the glass tube passes; when dry, distilled water is run in up to the mark, and the operation proceeded with in the ordinary manner: by this plan the possible error due to the variation of the surface tension is reduced to a minimum.

Estimation of Copper as Iodide.—M. Willenz.—Will be inserted in full.

Calibration of Flasks by Weighing.—A. Démichel.—The author gives a number of equations and formulae for calibrating flasks, which he claims will considerably simplify the operation.

A Tabular Atlas of the Chemistry of the Metals is being prepared by Mr. John Freemont Sleeper. It comprises the arrangement on a novel plan in sectional and tabular form, devised to rapidly convey information of the data relating to the history, distribution, properties, &c., of the metals of the alkalies, caesium, rubidium, and potassium. The author has been preparing this work for the past ten years, and hopes to have it published before long.

MISCELLANEOUS.

The Goldsmiths' Institute, New Cross.—A course of 25 lectures on "Coal Tar Distillation," to be given on Wednesday evenings at 8.30 p.m., by Mr. W. J. Pope, was commenced on the 27th inst. The course comprises the determination of boiling-points, the latent heat of vapours, specific heat of liquids; the composition and valuation of tars; plant and methods used: separation and purification of benzene, toluene, xylene, &c.; preparation of anthracene, carbazole, pyridene, &c., &c. Special attention will be paid to methods of analysis and to the plant used in this country and abroad.

Salicylic Acid and Calcium Sulphite as Preservatives of Cider.—Salicylic acid was unanimously condemned in 1882 as a preservative of beer, and very little can be inferred as to the physiological effects of its continuous use. Kolbe took a daily dose of it for over a year, increasing from 0.5 grm. to 1.5 grm., without any noticeable effect. On the other hand, a case is recorded where 48 grains caused death in forty hours. Salicylic acid is easily detected; the sensitiveness of the ferric chloride test has been put as high as 1 part in 100,000. Dr. A. B. Griffiths found that a 1/5000th solution of salicylic acid had no action on the true alcoholic ferment. Messrs. E. Bailey and Chas. Palmer have made a series of experiments with various strengths of salicylic acid on cider. Six flasks of cider were used containing salicylic acid of the following strengths:—1/20,000th, 1/10,000th, 1/5000th, 1/1000th, 1/500th, and a blank. Distillations were made after twenty-four hours, seventy-two hours, eight days, twenty-four days, and fifty-two days. Though the effect of the preservative is not very marked till a 1/1000th solution is used, yet it does seem that a 1/5000th solution has a noticeable effect upon the alcoholic ferments, and the micoderma seems to do very well in a 1/1000th solution. The action of sulphurous acid and its salts as preservatives has not been studied to the same extent, though it is known to be an active germicide. Its detection in small quantities is difficult, but in quantities sufficient to exercise any preservative action, sulphurous acid may be readily detected by zinc and HCl. Experiments on the effects of various amounts of calcium sulphite on cider show that the action is retarding only, for a considerable amount of alcohol is produced after the fifty-second day, even in a 1/250th solution.

Histological and Chemical Study of the Action of Antiseptics on Muscular Fibres.—A. Riche.—Continuing his paper from the last number, the author describes further experiments on the albumen and juice of meat, and he comes to the conclusion that sulphurous acid and the bisulphites—notably that of lime—alter the normal structure of the meat, and that the muscular fibre does not remain intact under their action, even at ordinary temperatures; that the soluble albumenoid matters do not behave in the same manner as in the presence of water, even when subjected to a temperature below 100°, or even at 50°. For these and other reasons it is not to antiseptics, but to refrigeration, that we must look to solve the problem of the preservation of meat.—*Journal de Pharmacie et Chimie*, Series 6, vol. vi., No. 5.

On a New Alkaloid.—MM. Battandier and T. Malosse.—The authors have extracted a perfectly definite alkaloid from the young roots and from the bark of the *Retama sphaerocarpa*; they have given it the name *Retamine*. One kilogram. of the plant gave 4 grms. of the alkaloid. A number of its properties are here given. It melts at 162°; it possesses extremely energetic reducing properties, &c.; chloride of gold and phospho-molybdic acid are instantly reduced, but salts of silver and ferricyanide of potassium more slowly. The salts of retamine crystallise very easily and with great sharpness, with the exception of the nitrate, which has up till now only been

obtained as a varnish. The average of eight analyses comparing the carbon and hydrogen, and of twelve of the nitrogen, give it the formula $C_{15}H_{26}N_2O$. Retamine is therefore an oxy-sparteine, but different to any other one known.—*J. de Pharm. et Chim.*, Series 6, vol. vi., No. 6.

MEETINGS FOR THE WEEK.

MONDAY, Nov. 1st.—Royal Institution, 5. General Monthly Meeting. Society of Chemical Industry, 8. "The Adulteration of Portland Cement," by W. H. Stanger, M.I.C.E., and Bertram Blount, F.I.C. "An Improved Adjustable Drip-proof Bunsen," by W. P. Evans, M.A., Ph.D.

WEDNESDAY, 3rd.—Society of Public Analysts, 8. "Estimation of Acetates in the presence of Inorganic Salts," by Bertram Blount. "Estimation of Carbonic Acid in Natural Waters," by C. A. Seyler, B.Sc. "Detection of Gelatin in Cream," "A New Milk Preservative," "A New Milk Adulterant," by A. W. Stokes. "Note on the Graduation of Lefman-Beam Bottles," by G. E. Scott Smith and A. B. Searle. "An Improved Milk Scale," by H. Droop Richmond.

THURSDAY, 4th.—Chemical, 8. "Properties of Liquid Fluorine," by Professors H. Moissan and J. Dewar. "Liquefaction of Air and the Detection of Impurities," "Absorption of Hydrogen by Palladium at High Temperatures and Pressures," by Prof. Dewar.

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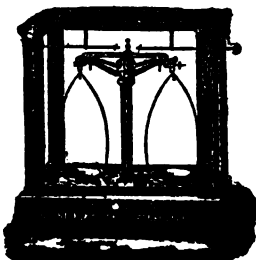
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FIG. 11. No. 91.

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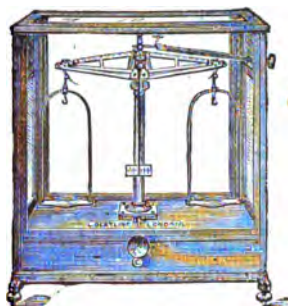
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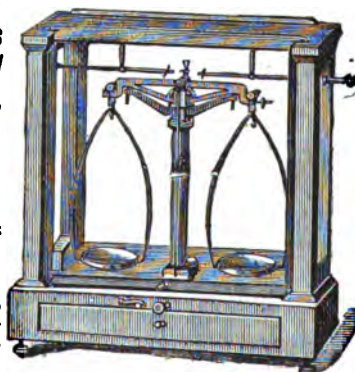


FIG. 3a.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1980.

DISTRIBUTION OF TITANIC OXIDE UPON THE SURFACE OF THE EARTH.*

By F. P. DUNNINGTON, F.C.S.

So far as I know, the discovery of the universal distribution of titanic oxide in the soil over the surface of the earth originated in the analysis of a cinder-like mass found about four miles from the University of Virginia and about a mile from "Monticello," the house of Thos. Jefferson being upon the slope of the same mountain.

This mass proved to contain 5.4 per cent of titanic oxide, and subsequently, upon analysis, to be of identical composition with the soil upon which it was found. The most probable suggestion of its formation is that it is a result of lightning. Other soil in that neighbourhood was examined for titanic acid, in all of which it was found; and subsequently, as many as eighty specimens of soil from various quarters of the globe. An account of these results was published by the author in the *American Journal of Science* for December, 1891.

It may be well here to give the averages for certain of these determinations:—

Titanic Acid in Soils. Averages of Determinations made.

					Percentage.
United States (40)	..	..	..	..	0.77
Great Britain (7)	..	..	..	..	0.75
Europe (10)	..	..	..	..	0.42
Igneous rocks—					
Germany (8)	..	..	..	..	0.56
Asia (8)	..	..	..	..	0.71
Japan (2)	..	..	..	..	0.55
Total average	..	..	..	..	0.63
Sandwich Islands (5)	..	..	..	..	3.17

It appears strange that any body existing in so considerable an amount in the soil should have been so constantly overlooked in the numerous analyses of soils that have been made; but this is in part to be accounted for by the fact that, for the purposes for which soil analyses are usually made, titanic oxide is considered as acting like alumina, and a distinction between them is regarded as a matter of little import.

This oversight is no doubt in part also due to the somewhat troublesome and unsatisfactory nature of the process of detecting and estimating this element which was employed prior to 1882, when Prof. A. Weller published in the *Berichte d. Chem. Gesell.* (xv., 2592) a method based upon the intense yellow colour of titanium trioxide, which is produced upon the addition of hydrogen peroxide to a solution of titanic sulphate.

Since publishing the foregoing results, I have been able to secure samples of soil from portions of the earth's surface not then represented; in these also the amounts of titanic oxide have been estimated as a percentage of the original soil and also of the ignited soil, which latter figures should be compared with what is found in igneous rocks. For a few of these determinations (which were made in duplicate) I am indebted to some of the students working in the Chemical Laboratory at the University of Virginia; those of the deep well borings were made by Mr. David Hancock, of Virginia.

* Read before the British Association (Section B), Toronto Meeting, 1897.

- No. 81.—Red loam; and No. 82, dark grey loam; Brazil.
 „ 83.—Grey sandy loam, Liberia; per Mr. Apperson at the Chicago Exposition.
 „ 84.—Grey brown loam, St. Helena; per Professor Cleveland Abbe, Washington, D.C.
 „ 85.—Dark grey clay; and No. 86, light brown clay, Cerraloo, Mexico; per Mr. J. T. De Bell.
 „ 87.—A light brown clay; and No. 88, white clay, Sydney, Australia; per Prof. A. Liversidge.
 „ 89.—Brown-yellow loam, Launceston, Tasmania; and No. 90, brown-grey loam, Auckland, New Zealand; per Mr. H. J. Boyd.
 „ 91.—Light yellow clay, Araki, Japan; and No. 92, dark grey loam, Kawaja, Japan; per Mr. Apperson.
 „ 93.—Fine brown and green gravel, from earthquake eruption, Nayaya, Japan; per Rev. R. B. Grinnan.

(Nos. 94 to 104, from Dominion of Canada, were sent by Dr. G. M. Dawson, of Ottawa).

- No. 94.—Pale yellow fine sand; Champlain, Quebec.
 „ 95.—Brown loam; Ottawa.
 „ 96.—Grey clay; near St. Luis Dam, Ottawa.
 „ 97.—Brown loam; Beechwood, Ottawa.
 „ 98.—Pink loam; Murillo, Algomo.
 „ 99.—Black earth; Rosser, Manitoba.
 „ 100.—Fine black sand; MacGregor, Manitoba.
 „ 101.—Fine grey siliceous sand; Maple Creek, Assiniboia.
 „ 102.—Dark grey sand; Canmore, Alberta.
 „ 103.—Light brown clay; Griffin Lake, B.C.
 „ 104.—Light red loam; Glacier, B.C.

(Nos. 105 to 114, drillings from the deep well at Wheeling, W. Va., obtained through Prof. Wm. Halleck, then at the Physical Laboratory of the U.S. Geological Survey).

The percentage of titanic acid found in these respectively is as follows:—

No.	Air-dried.	Ignited.	No.	Air-dried.	Ignited.
81.	1.33	1.79	93.	0.49	0.48
82.	0.74	0.83	94.	0.34	0.41
83.	0.62	0.66	95.	0.68	1.37
84.	2.33	3.00	96.	0.68	0.74
85.	0.22	0.25	97.	0.61	0.67
86.	0.14	0.20	98.	0.30	0.32
87.	0.69	0.79	99.	0.64	0.79
88.	0.43	0.48	100.	0.22	0.24
89.	0.50	0.61	101.	0.22	0.23
90.	1.40	1.57	102.	0.18	0.25
91.	0.52	0.55	103.	0.17	0.17
92.	0.43	0.46	104.	0.52	0.54

No.	From depth of—	Air-dried.	Ignited.
105.	275 feet	0.09	0.13
106.	500 „	0.79	0.89
107.	992 „	0.12	0.13
108.	1460 „	0.12	0.13
109.	2010 „	0.74	0.77
110.	2520 „	0.35	0.36
111.	3075 „	0.80	0.85
112.	3500 „	0.77	0.82
113.	4050 „	0.49	0.53
114.	4490 „	0.48	0.51

Average.. .. 0.475 0.512

These facts point plainly to the universal distribution of this element over the earth's crust. I would draw attention to the figure 0.56 per cent of titanic acid from the alluvial soil of the Yellow River in China, as determined in the former set of observations, as most probably presenting a fair average of the amount present upon the earth's surface.

For all the above figures the total averages are 0.57 and 0.66. Omitting the Island of St. Helena, as exceptional, we have 0.575 and 0.588 respectively for the air-dried and ignited soils.

University of Virginia, August, 1897.

SEPARATIONS WITH ALKALINE ACETATES.

By HARRY BREARLEY.

(Concluded from p. 211).

VI. Zinc from Iron. General Considerations.

ZINC and manganese are the only two metals whose acetate separation from iron is so far favoured as to be commonly employed. In the case of Zn the fault is again the operators: carried out in the previously reiterated manner, the separation is one of the easiest and most complete of the series.

Two tests were made on solutions of 1 grm. iron and 0.1 grm. zinc. The zinc was estimated by precipitating as carbonate, and, so as to balance any errors, the value of the stock zinc solution was estimated under like conditions. The separations were carried out as before.

Acetate used.	Zinc recovered.
20 c.c.	0.1008 grm.
50 "	0.0976 "

No further observations on the separation of iron and zinc were made. If it be protested that such scant attention to zinc is by no means commensurate with its importance, it should be remembered that these papers are written from the standpoint of a Steel-works Analyst, who must make them as far as possible of momentary utility. In this light zinc is not an important element in combination with iron, and its separation therefrom is relatively of little value. It is by no means overlooked that in the larger world of analytical chemistry the separation is an important one, and therefore the tests were made with a view of allocating the element in the table of relative separability, if I may so term it, of the elements treated. There is, moreover, independent testimony to the claim of zinc to occupy its allotted place, in the fact that Mr. Jewett—whose paper I am unable to refer to—has found, in a somewhat different way, that manganese and zinc could be completely separated from iron by the acetate process, but not so nickel or cobalt.

The above defect is not really a very serious one. The influence of a particular variation can be readily made by anyone specially interested in the separation. It may be noticed, too, by those practically acquainted with the separation and estimation of the metals in Table XIX., that their relative behaviour with increasing acetate is a reliable indication of the liberties that may be taken with the processes without incurring the penalty of an imperfect separation.

And now, so far as I am aware, every metal whose acetate separation from iron is practised has been dealt with. There are, it is true, the alkaline earths; and as they are, more or less, always to be found in iron ores, slags, &c., they should not be omitted without reason. Good enough reason is to be found in the fact that the separation has long ago been established as a perfect one, no writer in standard books ever doubting its accuracy so far as to recommend a re-solution and precipitation of the basic ferric acetate.

A noteworthy point, not strictly within the scheme, is the behaviour of phosphoric acid. This compound invariably goes down with the iron in an ordinary acetate precipitation. Three tests were made to detect any possible variation from this course. Each test solution was made up of 1 grm. of iron and about $\frac{1}{4}$ a grm. of soda phosphate.

- I. contained total hydrate and 10 c.c. acetic acid.
- II. " " 30 " "
- III. " no hydrate, and no free acid other than 30 c.c. acetic.

The iron was precipitated in each with minimum acetate. The filtrates were boiled to dryness; the residue of soda salts ignited, re-dissolved in dilute hydrochloric, neutralised with ammonia, acidified slightly with nitric acid, treated with the usual molybdate reagent, and placed side by side in a warm place. In I. there was only the faintest trace of a precipitate visible, and that chiefly where the rod had rubbed against the beaker. In II. there was somewhat more, but not enough to make any material difference to an analysis conducted in this way. In III. there was a very decided precipitate; such a quantity as makes a very serious difference when regarded as a deficiency. The amount was not determined quantitatively, it being rather the purpose of these remarks to guard against any possible error than to state definitely how great a percentage recovery is possible by these means.

The accompanying table (XIX.) affords perhaps the best general summary that could be given. It is only necessary to point out that each separation was made from a solution containing 1 grm. of iron and 0.1 grm. of the respective metal. Total hydrate was formed in the solution, and 10 c.c. acetic acid added. The volume of the solution was 1 litre. In some cases soda salts have been used, in others ammonia. This is the only difference in the treatment of the respective elements, and does not, I believe, involve in any case a change in the percentage recovery large enough to give any element precedence of its neighbour.

It was not practicable to include in Table XIX. the turbidity temperatures, although they afford the best proof I can give that the separations were made under comparable conditions.

The temperature at which a turbidity forms seems to have been generally overlooked. The observations are so readily made, and such reliable accompaniments of better or worse separations, according to their value and general conditions of working, that I venture to recommend the use of a thermometer for confirmatory purposes whenever a large number of estimations are made rapidly after the manner here set forth.

Wollcott Gibbs (CHEMICAL NEWS, xi., 102) has pointed out that the acetate should be added to the solution when cold, and then heated to boiling. This precaution has been repeated by Crookes ("Select Methods," p. 227, 3rd edit.), by Arnold "Steel Works Analysis," p. 166), and others. Its advantage, when large excesses of acetate are used, is not questioned, otherwise the precaution becomes an unnecessary time absorber.

The fact that a perfect separation of Mn, Ni, &c., may be made by adding dilute acetate to the boiling solution, is one that in every-day practice has been regularly made use of. In dissolving the sample of (say) nickel steel, an approximately equal amount of acid is always used, and so one has some idea of the volume of alkali needed to effect neutralisation. Something less than this amount may be added at once, and then the neutralisation completed by adding in rapid succession several c.c. of the carbonate at a time. It is really unnecessary to wait for all traces of the preceding turbidity to disappear, because finally an addition of carbonate will cause a precipitate altogether unlike the preceding ones. Though hard to describe, the difference is easy to distinguish. The final precipitate is thicker, and pulls irregularly on the hand as the flask is whirled. In small volumes of solution it is almost like a jelly. This is an unmistakably permanent turbidity, produced almost as quickly as the description may be read, and not by any great excess of alkali either. To this turbid solution add 10 to 12 c.c. acetic acid. The turbidity quickly disappears, part of the acid going to form acetate. Dilute with hot water, heat fur-

TABLE XIX.
Percentage separation from iron of—

Dilute acetate. C.c.	Mn.	Zn.	Co.	Ni.	Cu.	Cr as CrO ₃ .	Cr as Cr ₂ O ₃ .	Al.
10	100.0	—	100.0	100.0	98.0	26.8	trace	Precipitation
20	100.0	100.8	99.0	99.0	92.9	30.8	(See p. 175).	of iron
50	100.0	97.6	97.5	95.2	69.6	38.8		not possible
100	100.0	—	93.8	90.0	53.1	47.5		(see p. 210).

SYNOPSIS.

Metal.	Acetate.	Oxide.
Manganese	Crystallises in plates; permanent in air..	Easily soluble in acids. Readily soluble in (NH ₄)Cl aq.
Zinc	Crystals may be sublimed as zinc acetate	Easily soluble in acids. Soluble in hot solutions of ammonium salts.
Cobalt	Crystallises; red needles	Easily soluble in acids; only slowly when cold. Soluble in hot NH ₄ Cl.
Nickel	Apple-green prisms	Insoluble in acetic acid (!) Only slightly soluble in hot NH ₄ Cl.
Copper.. .. .	Dark green crystals. There are also sesqui-, bi-, and tri-basic cupric acetates. These form when Cu is exposed in contact with acetic acid, as in common verdigris ..	Soluble in acids; slowly in NH ₄ salts. Soluble in boiling H ₂ O solutions of Al ₂ , Cr ₂ , Fe ₂ , with precipitation of oxides of the bases of these salts. Unacted upon by boiling solutions of Mn, Ni, Co, Zn.*
Chromic acid	The deficiency is due to an altogether different cause. The percentage recovery increases directly as the acetate. Acetic acid not notably advantageous.	
Aluminium	Decomposes on evaporation. There is a soluble basic acetate; but if this is heated or left to evaporate at ordinary temperature it deposits insoluble basic salts	

The relative separation of an element varies directly as the stability of the acetate and the solubility of the oxide under experimental conditions. The general behaviour of the acetates and oxides in the above synopsis is in accord with this statement.

* This division is exactly that Cu makes in the Table. The active below; the inactive above.

ther, and, if the solution does not become turbid about 90° C. (it should never be turbid below 80° unless large amounts of alkaline salts are present), a few c.c. of dilute acetate should be added. The process may have been clumsily pictured, but it has been practised scores of times, and in experienced hands renders the estimation of nickel in steel the most rapid of all laboratory estimations save colour carbon.

The addition of free acetic acid is not a new idea. "A few drops to prevent the formation of basic acetate of the protoxide" is a familiar injunction. Phillips ("Engineering Chemistry") adds 2 c.c. of 10 E HCl after just dissolving the permanent turbidity. Eggertz (CHEMICAL NEWS, xviii., 232) adds 1½ c.c. HCl. I believe that Jewett gives other and earlier examples in his paper. These additions of HCl serve a good purpose when large amounts of acetate are used, in that they provide more ferric chloride to be decomposed, and leave a smaller excess of acetate to act undesirably on the Mn, Ni, or Co, as the case may be.

The addition of HCl or HNO₃ to a hydrate-free solution finally resolves itself into the same thing as adding free acetic acid, because, owing to their superior avidity, they react so,—



and a similar change takes place when, through added hydrochloric acid, the proportion of ferric chloride is increased. These theoretical considerations may be verified by noticing that it is indifferent to the turbidity temperature and the accuracy of a separation whether—having a solution containing a known amount of free acid—it be treated by neutralising and adding acetate and acetic acid, or by reversing the order in which the reagents are added, or by mixing them up in any way whatever.

I am persuaded to indulge in the following speculations by the fact that the only author (J. O. Arnold, "Steel Works Analysis," Whittaker and Co., 1895) who makes a special feature of explaining the processes of steel analysis dismisses the acetate separation with "On the addition of ammonium acetate the iron is precipitated as an indefinitely constituted mixture of ferric acetate and hydrate generally known as basic ferric acetate, and some free acetic acid is liberated. The complex reactions bringing about this result are not clearly known." I am encouraged also by the hope that if I miss the mark, which is very possible, I may hit some one else into providing a better explanation.

The precipitation of a solution of ferric chloride may be represented so,—



and previous experiment has shown that the proportion of Fe₂Cl₆ and NaC₂H₃O₂ required to effect the precipitation are in fair accord with the equation's requirements.

A solution of ferric acetate on heating is precipitated. The temperature at which precipitation takes place has been shown to be moderately constant. An excess of acetate, however, above that necessary to decompose the ferric chloride effects a separation at a lower temperature, and this decreasing of the temperature takes place in a quite regular manner for increasing acetate.

The precipitated ferric acetate is not a very stable body. In the boiling solution it becomes converted into ferric oxide and free acetic acid. This I surmise is the free acetic acid Prof. Arnold refers to, though I do not know that its liberation is necessarily concurrent with the precipitation, which may take place at very low temperatures. The volume of acetic acid formed is proportional to the

amount of ferric acetate, that is to the amount of ferric chloride previously unchanged into dissolved ferric hydrate by the neutralisation. This explains why, in the copper separations, the solutions free from dissolved ferric hydrate behaved similarly to those having large amounts of acetic acid present. It also provides another reason why the addition of a few c.c. of HCl, as practised by Eggertz, Philips, &c., lead to a more accurate separation. Plainly, then, the composition of the "basic acetate" will depend on the proportion of dissolved hydrate—which is dehydrated in the boiling solution—and ferric chloride in the first instance; and then on the extent to which the ferric acetate and hydrate are decomposed by heating. Thus the composition of the precipitate must always be the indefinite—



but the reactions by which it is formed seems to be as understandable as reactions generally are.

With some misgivings I go a step further, and attempt to explain the imperfect separation and the relative positions of the elements in Table XIX. It is remarkable that with the exception of copper, the elements are all members of the iron group; indeed they constitute the whole group.

The explanations required are why an increasing excess of acetate gives a decreasing separation, and why the addition of acetic acid arrests this defection.

Having seen that an addition of soda acetate to a solution of ferric chloride forms ferric acetate, there is no difficulty in understanding that a similar change—and the more readily effected the larger the proportion of alkali acetate—can occur with manganese, zinc, cobalt, &c.

When heated the ferric acetate is precipitated. A similar course is not followed by the other metallic acetates, at least not in pure solutions. Heating at boiling temperatures decomposes ferric acetate in the manner previously mentioned, and through an analogous behaviour of the acetates of the other metals the defective recovery may be accounted for.

I find, on turning to Watts' "Dictionary of Chemistry" (vol. i., p. 9), that "many acetates may be decomposed by water into acetic acid and metallic oxide." This decomposition in the case of aluminic and ferric acetates occurs at 100°, while at 175° the acetates of Mn, Co, Ni, Zn, and Cu are slowly decomposed.

It is true that the acetate separation never involves a temperature beyond 103° to 104° C., and that a defective separation may be obtained at much lower temperatures than that even (60° to 70°), if enough acetate has been used. It is also true that what takes place in a solution of pure salt is frequently modified when the same salt acts in a mixture. I admit the presumption, and yet I beg to be allowed, tentatively at least, to state that these metallic acetates, in company with the iron, are decomposed at a much lower temperature. The evidence, then, for framing a palpable explanation to the first query is complete.

The solubility of one of the products of the decomposition—the metallic oxide—in dilute acids is a sufficient explanation of the action of acetic acid.

If these explanations are true ones we should be able to justify, in an independent manner, viz., by the behaviour of their acetates and oxides, the position of the elements in Table XIX. This I believe we may do fairly satisfactorily.

Perhaps the most effective way will be to let a short Synopsis speak for itself (see p. 223). The data are taken from Watts' "Dictionary" and Comey's "Solubilities." The action of alkali chlorides, which have been occasionally noticed, are also explained.

It is hoped that the foregoing papers may do something to restore alkaline acetates to the favour they deserve.

Attention was drawn months ago to the fact that ammonium acetate was said to be unstable and needed to be

made as required. It may not be amiss to say that the solution then in use is being used now, and so far as I can detect in the working is quite unaltered.

In conclusion, I most warmly tender my thanks to Mr. R. L. Leffler and Mr. Jervis, respectively chemist and co-assistant in this laboratory, for their general interest and appreciated aid in the work.

The Laboratory,
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Norfolk Works, Sheffield.

ON CARMINIC ACID.

By W. VON MILLER and ROHDE.

CARMINIC acid, dried *in vacuo* at an ordinary temperature, yielded on combustion with lead chromate:—

Carbon	53.65
Hydrogen	4.32

Carminic acid dried in a stream of hydrogen at 115° to 120°:—

Carbon	53.77
Hydrogen	4.27
(Burnt with copper oxide).	

Carminic acid dried *in vacuo* at 80°:—

Carbon I.	53.68
" II.	53.80
" III.	53.75
Hydrogen I.	4.5
" II.	4.7
" III.	4.4

(All burnt with lead chromate).

A sample of crystallised acid prepared according to the procedure of Schunck and Menschutkin gave similar values dried in a current of hydrogen at 115°:—

Carbon	53.69
Hydrogen	4.24

Berichte der Deut. Chem. Gesell., No. 13, p. 1759.

ON THE COMPOSITION OF CERTAIN CANADIAN VIRGIN SOILS.*

By FRANK T. SHUTT, M.A. F.I.C., F.C.S.,
Chemist, Dominion Experimental Farms.

(Concluded from p. 210).

The Maritime Provinces.

THE soils from New Brunswick and Nova Scotia examined by us have been so few in number that it would be unwise to draw from the data conclusions as to the general character of the soils of these provinces. A few examples are here given which, though representative of large areas, must not be considered as the only provincial types; the figures are inserted here to render the data somewhat more complete than they otherwise would be. (See Table VI.).

New Brunswick.

Soil No. 57.—From the Sackville Marsh, at the head of the Bay of Fundy. A clay loam; of interest as an example of a soil area very uniform in character—a fact no doubt due to the origin of the soil, which is practically a tidal deposit. When thoroughly drained, which frees them from salt and improves their texture, these reclaimed Marsh soils are found to be exceedingly fertile. A glance at the

* Read before the British Association (Section B), Toronto Meeting, 1897.

TABLE VI.—Analyses of Soils (Water-free), The Maritime Provinces.

No.	Locality.	Surface or subsoil.	Character of soil.	Potash.	Phosphoric acid.	Nitrogen.	Lime.	Loss on ignition (organic and volatile matter).
57.	Sackville Marsh, N.B.	Surface	Clay loam	0.16	0.16	0.131	0.13	5.83
58.	Restigouche, N.B.	"	Yellow sandy soil	1.02	0.10	0.113	0.23	5.46
59.	Cumberland, N.S.	"	Sandy loam	0.16	0.09	0.090	0.06	3.37
60.	S.W. Mabou, N.S.	"	"	0.37	0.09	0.212	0.05	6.97
61.	King's Co., P.E.I.	"	"	0.47	0.09	0.106	0.08	5.10

analytical data shows that this is not to be altogether ascribed to large percentages of plant food, and it is more than probable that the fine state of division and the intimate incorporation of the soil particles—due to the manner of the soils formation and deposit—render the elements of fertility more easily obtained and assimilated by the plant.

Soil No. 58.—Balmoral Settlement, Restigouche. A yellow loam, derived principally from the decomposition of felspar, though showing some quartz fragments. The percentage of potash is considerably above that found in average fertile soils—a fact undoubtedly due to the felspathic origin of the soil. With the exception of potash, however, the soil cannot be considered one equal to Canadian soils of average fertility.

Nova Scotia.

Soil No. 59.—A reddish sandy soil from Hansford, Cumberland Co. It is below the average in the more important elements, and to be regarded as a poor soil, but responding well to judicious culture and manuring.

Soil No. 60.—A soil from South-west Mabou, Inverness; very similar in appearance to No. 59, but analysis shows it to be much richer. The small percentage of lime is particularly noticeable in these soils; the knowledge of this fact has assisted towards the economical treatment of them with fertilisers.

Prince Edward Island.

Soil No. 61.—This soil partakes of the same colour as the light red Triassic sandstone from which it is derived, and in this respect at least this sample is representative of the characteristic soil of the province. It differs from the preceding specimens in that it is not a truly virgin soil. Some difficulty was experienced in procuring a sample which had not been cropped or manured; indeed, no guarantee of such could be obtained. This soil, however, is said to fairly represent the unmanured but cultivated soil that extends over a large area in the Eastern portion of the Island. It is light sandy loam, the texture of which is fairly good. Though containing a more than average amount of potash, this soil could not be ranked, from a chemical standpoint, with our richer Canadian soils—possessing but small percentages of nitrogen, phosphoric acid, and lime.

This agricultural province is justly known as a fertile one; and we presume, judging from such data as we have at hand, that this fertility is due rather to good soil texture and favourable climatic influences than to richness of its land in plant food constituents.

The last table (Table VII.) that is presented for your consideration, showing the average amounts of fertilising ingredients in the surface soils that have been examined, taken province by province, has been prepared with no little diffidence. If it were to be interpreted as placing before you data from which deductions could be made as to the average soil fertility of the yet untilled areas of the respective provinces, it might be regarded as misleading. It is not my intention that such a conclusion should be drawn. A hundred or so samples, though they are typical, and, as far as possible, thoroughly representative of large areas, taken from the thousands of square miles of untilled soil in the Dominion, do not afford sufficient basis for such generalisations. They are not provincial averages; they are rather averages from large un-

tilled areas in the several provinces, and may therefore serve to indicate the general character of much of the yet untilled lands in Canada.

TABLE VII.—Analyses of Soils. Averages. Surface Soils.

No. of samples.	Province.	Potash.	Phosphoric acid.	Nitrogen.	Lime.
21	British Columbia	0.42	0.27	0.262	1.17
7	North-West Territories and Manitoba	0.44	0.19	0.537	1.08
6	Ontario (Muskoka only)	0.22	0.15	0.135	0.44
6	Quebec	0.44	0.20	0.226	0.52
5	Maritime Provinces	0.44	0.11	0.130	0.11
45	Average of all..	0.39	0.18	0.258	0.66

When we remember that care and judgment were exercised in the selection and collection of these samples, that the analyses were carefully conducted according to modern and approved methods, that very few of the individual samples fall below the standards or limits fixed by agricultural chemists, and that many contained such ample stores of plant food as to warrant them in being classed among the most fertile soils, we may, I think, safely conclude that the data here set forth clearly indicate that while there are many types of soils represented in Canada, there are in all her provinces large tracts of land that, as far as plant food is concerned, compare favourably with the most productive of other countries.

Canada is fast becoming known in the markets of the world as a great food-producing country. Soils rich in plant food and favourable climatic influences are the chief factors that have assisted the Canadian agriculturist in building up this reputation. These are the factors, together with intelligent rational methods of farming and safe cheap means of transportation, that will continue in the future to make agriculture here a prosperous industry. It is therefore gratifying to learn that ample scientific proof is now on record to show that in our virgin soils there is in such abundance the crude materials upon which crops must directly, and farm animals indirectly, thrive.

EARLY AMERICAN CHEMICAL SOCIETIES.*

By Prof. H. CARRINGTON BOLTON.

(Continued from p. 217).

II. The Columbian Chemical Society of Philadelphia.

The Columbian Chemical Society was founded in the month of August, 1811, by "a number of persons desirous of cultivating chemical science and promoting the state of philosophical inquiry." The names of the gentlemen who attended this meeting are not certainly known, but it may be presumed that they included most of those who were then elected to office; these were as follows:—

* Read before the Washington Chemical Society, April 8, 1897. From the *Journal of the American Chemical Society*, August, 1897.

Patron—Hon. Thomas Jefferson, Esq.

President—Prof. James Cutbush.

Vice-Presidents—George F. Lehman and Franklin Bache.

Secretary—John C. Heberton.

Treasurer—James J. Hamm.

Orator—John R. Barnhill.

And a "Corresponding Committee" of Three: John Barnes, M.D.; John Lynn, M.D.; and Charles Edwards.

Thomas Jefferson's commanding position in the world of science and arts, as well as his literary attainments, well qualified him for the dignified office of patron. He had held the office of president of the most prominent scientific body in the United States (American Philosophical Society) for many years, and only relinquished it to accept the higher one of Chief Magistrate of the Nation. Seventeen months before the founding of the Chemical Society, Jefferson had retired from the presidency, after serving his country eight years, and was living at his country seat, Monticello.

James Cutbush, president of the Columbian Society, was at that time professor of natural philosophy, chemistry, and mineralogy at St. John's College. Little is known of his early history: in 1814 he was appointed to the army with rank of Assistant Apothecary General, and he held the position of chief medical officer of the United States Military Academy at West Point, from June, 1820, to November, 1821; the army being re-organised, he became assistant surgeon and acting professor of chemistry and mineralogy at the same institution, positions which he held until his death, December 15, 1823.

Dr. Cutbush's papers, presented to the Columbian Society, will be considered below. He published also the following:—"On the Formation of Cyanogen in some Chemical Processes not before noted" (*Am. J. Sci.*, vi., 1822), "On the Composition and Properties of the Chinese Fire" (*Ibid.*, vii., 1823), "On the Composition and Properties of Greek Fire" (*Ibid.*, vi., 1822). He was also the author of several books:—"Useful Cabinet" (1808), "Philosophy of Experimental Chemistry" (Philadelphia, 1813), and "A System of Pyrotechny" (Philadelphia, 1825). The last-named is an elaborate work of more than 600 pages, octavo.

George F. Lehman, the first vice-president, published articles in Mitchell's *Medical Repository*, chiefly on medical subjects.

Franklin Bache, the second vice-president, was at that date a youth of only twenty years, who had graduated at the University of Pennsylvania the year before the founding of the society. He was a grandson of Benjamin Franklin, and a member of the distinguished Bache family, which numbered so many eminent men of science. He afterwards became professor of chemistry at the Franklin Institute, and in 1841 at the Jefferson Medical College, which chair he held until his death in 1864. He is remembered also as the author of "A System of Chemistry for the Use of Students of Medicine" (Philadelphia, 1819), and of other chemical treatises.

The constitution adopted by the founders of the society, besides the usual provisions for regulating business, contained some unusual features; the officers included an orator, and Article VII. prescribed:—"An oration on some chemical subject within two months after the commencement of the medical lectures in the University of Pennsylvania, in each year." Since the "Memoirs" published by the society contain no "oration," it is to be feared that the incumbent's efforts were not satisfactory.

Two articles in the constitution deal with fines:—"Every member shall be fined 12½ cents for absence each roll, unless satisfactory reasons be offered." And again, "Any member being elected to office and refusing to serve shall be fined one dollar."

Another notable provision is as follows:—"The society shall appoint, once in each month, some member to read an original chemical essay, for neglect of which the member so appointed shall be fined one dollar." These

fines, with the annual fee of two dollars, were evidently expected to maintain a full treasury.

To become a member of the society special qualifications were prescribed; after being proposed and seconded the candidate "shall read an original essay on some chemical subject, on which any member may speak not more than ten minutes." After this trial of his ability, a two-thirds vote of the members present at a subsequent meeting were required to secure election.

It seems to have been easier to be put out of the society than it was to get in, for "any member behaving in a disorderly manner shall be expelled by consent of two-thirds of the members present."

This mandatory "shall" is used throughout the regulations; the president "shall preserve order," the secretary "shall keep fair minutes," the constitution "shall be revised annually," and so on. To insure against members withdrawing early from a dull meeting, the "secretary shall call the roll at the opening and close of each meeting and mark down absentees," each of whom is then fined 12½ cents as stated. Never did a society undertake to control its members with more stringent rules!

The members who subscribed to these regulations were divided into two classes, "Junior" and "Honorary" members, the former corresponding to a class which would now be styled "Associates," and the latter including both American and foreign chemists of distinction. The junior members numbered thirteen, the honorary members numbered sixty-nine, thirty-one of whom were Europeans. The home list included most of those chemists, then living in America, whose labours contributed largely to the foundations of the science in the New World. Brief notices of some of the members will serve to summarise the status of chemistry in the United States for the years 1811 to 1813.

Dr. Benjamin Smith Barton (1766-1815) held the chair of medicine, natural history, and botany in the University of Pennsylvania. Dr. Barton has been called by his admirers "the father of American natural history," though there are other claimants for this honourable designation—Mitchill, of New York, and Thomas Jefferson. Dr. Browne Goode, writing of Barton, says he, of all the early Philadelphia naturalists, "had the most salutary influence on the progress of science." He was a leader in the American Philosophical Society, and an agreeable writer on natural history topics, and, though he made no contributions to chemistry, was a worthy member of the society.

Dr. Archibald Bruce (1777-1818), one of the pioneers of mineralogical science in America: he had established the *American Mineralogical Journal* one year before the date of which I write. His analyses of minerals mark him as a skilful chemist. He held the chair of mineralogy in Columbia College, New York.

Joseph Cloud (1770-1845) was assay master of the United States Mint in Philadelphia, and already distinguished by his researches on palladium (1807).

Thomas Cooper (1759-1840), born in London, had come to America in 1792, with his friend Priestley, whose radical views in politics and religion he shared. Dr. Cooper wrote much on political, ethical, and philosophical subjects, and published some essays on chemistry. In 1811-14, the period of the Columbian Chemical Society, he held the chair of chemistry at Dickinson College, Carlisle, Pa., and in 1819-34 he held the same position at the College in Columbia, S.C., of which he afterwards became president.

Dr. John Redman Coxe was professor in the medical department of the University of Pennsylvania, having succeeded Dr. Woodhouse. He made several original observations in chemistry published in current periodicals.

Dr. Edward Cutbush (1772-1843) was surgeon of the United States Navy and professor of chemistry in the medical school of the Columbian University, Washington (1825-27). He has another honourable claim to distinction, having been the founder in 1819 of the

Columbian Institute for the Promotion of Arts and Sciences in Washington, a sort of precursor of the Smithsonian Institution.

Passing with brief mention Dr. Elisha de Butts, professor of chemistry in the College of Maryland, Prof. Benjamin de Witt, of New York, and Dr. John Syng Dorsey, already mentioned as a member of the society founded in 1792, we reach the more familiar name of Dr. John Griscom, "the acknowledged head of all teachers of chemistry in New York City," for more than thirty years.

The next name in the alphabetical list of members is that of Robert Hare, professor of natural philosophy in the University of Pennsylvania, whose career we have already noticed.

Dr. David Hosack (1769-1835), professor of botany and materia medica in Columbia College, New York, is best known as the founder of the first public botanic garden in the United States, in 1801. His contributions to science were chiefly in medicine. The tragic circumstances of his death have been nearly forgotten; he died of shock at the disastrous conflagration in New York City in 1835, which swept away his property to the value of \$300,000.

Dr. Henry Jackson, professor of chemistry at Athens College, Georgia, is followed by His Excellency James Madison, LL.D., President of the United States of America, whose name added lustre to the rolls of the society, but whose claim to the membership can only be based on extensive general information.

Dr. John Manners, of Philadelphia, affixes to his name the initials F.A.N.S., the Academy of Natural Sciences, having been founded one year before the printing of the list of members. His contributions to the Chemical Society will be noted below.

Dr. John Maclean (1771-1840) was the first professor of chemistry in the College of New Jersey, now Princeton University, to which chair he was elected in 1797. In accordance with the prevailing custom, he also gave the instruction in astronomy, mathematics, natural philosophy, and natural history; this fact is ample apology for his not appearing in the ranks of original investigators. Prof. Maclean published in 1797 "Two Lectures on Combustion," in which he upheld the views of Lavoisier, as opposed to the phlogistic theory maintained by Dr. Priestley.

The Hon. Samuel L. Mitchell, M.D., F.R.S.E. (1764-1831), professor of chemistry and natural history in Columbia College from 1792, was active in many branches of scientific research. In 1798 he established the *New York Medical Repository*, which for sixteen years was an influential organ in recording and diffusing progress in general science, as well as in medicine. His zeal for science did not prevent his taking part in national affairs, for he occupied a seat in the senate of the United States from 1804.

Dr. Thomas D. Mitchell, F.A.N.S., was one of the most active members of the society, frequently contributing to its memoirs.

Passing by Dr. John C. Osborne, professor of the institutes and practice of medicine in Columbia College, New York; Dr. Joseph Parish, of Philadelphia; Mr. Robert Patterson (1743-1824), professor of mathematics and lecturer on natural philosophy in the University of Pennsylvania, director of the United States Mint, and afterwards (1819) president of the American Philosophical Society; Dr. Nathaniel Potter, professor of the theory and practice of medicine, University of Maryland, we reach the eminent Dr. Benjamin Rush, professor of the institutes and practice of medicine in the University of Pennsylvania. Dr. Rush (1745-1813) has been characterized by Benjamin Silliman as "undoubtedly the first Professor of Chemistry in America, his appointment dating August 1, 1769. In his busy life, besides his professorial chair, he filled the positions of surgeon-general of the United States Army (1777), treasurer of the Mint,

president of the Society for the Abolition of Slavery, vice-president of the Bible Society of Philadelphia, and conducted a large medical practice in the same city.

(To be continued).

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, October 29th, 1897.

Mr. SHELFORD BIDWELL, President, in the Chair.

PROF. STROUD exhibited and described the "*Barr and Stroud Range-finder*." The problem of finding the distance of a given object at sea, or in the field, is complicated by the shortness of the trigonometrical "base" and by restrictions of time. As a rule the apparatus must be self-contained, and "snap-shot" readings are obligatory, —i.e., the range has to be determined from a single instrument and from a single observation. At 3000 yards the errors must not exceed 3 per cent. In foggy weather, or when viewing a nebulous object, this degree of precision is difficult to attain; but under favourable circumstances the authors have determined ranges, at that distance, within 1 per cent of accuracy. At shorter ranges measurement is more exact; thus an object at about 2000 yards may be estimated to within about 12 yards. Prof. Stroud gave some account of the history and of the general methods employed in these instruments. Two images of the distant object, preferably of a line such as a flag-staff, are received respectively upon two mirrors, two lenses, or two prisms, placed one at each end of a fixed support. From each of these the light is then directed towards the middle of the instrument, where the two images, after further reflection, are viewed by one eyepiece. The optical system has finally to be adjusted so that the two images, as now seen in the eyepiece, lie in the same straight line. In the instrument designed by the authors this coincidence is attained by translating a small prism parallel to the axis of the supporting rod. The extent of this translation is a measure of the range. Both eyes are used; the right for bringing the two images into alignment; the left for "finding" the object through a small field-glass, and for reading the scale of distances. At night, sightings have to be taken from "points" of light, and, as these are unsuited to measurement, the authors convert them into "lines" by the use of cylindrical lenses. Various devices are introduced to prevent over-lapping of the images. The instrument is about 5 feet long and tubular in form; it is made of copper, so as to have high thermal conductivity to reduce differential heating. Within the outer tube is the interior supporting rod, designed to equalise so far as possible the effects of interior radiations. Several forms of "separating" prisms were exhibited: the best for the purpose consists of two "reflecting" prisms; these receive the two rays, and direct both of them into a third prism, whose angle lies in the space between the angles of the others.

Mr. BARR drew attention to the gimbal arrangement and the three struts that keep the supporting rod central in the tube. To give some idea of the precision and scope of the range-finder, he observed that they were there using the equivalent of a 25-ft. "circle," and their measurements were comparable to the measurement of 20 secs. of angle, on such a circle. The instrument is handled by ordinary seamen, and stands rough usage on board ship for years without injury.

Prof. STROUD then exhibited a "*Focometer and Spherometer*." He explained that in determining curvatures and focal lengths some telemetric method was necessary, and that, owing to want of parallelism of the beam and

duplication of images, a short-focus telescope was always an inefficient telemeter. For the measurement of inaccessible lengths it was therefore better to use some simple form of "range-finder." Such an apparatus could be made with a set of small mirrors, arranged in such a manner as to direct two images of the distant object into an eyepiece, with a fixed prism in the path of one of the incident beams. By sliding this instrument along the optical bench, one position could always be found at which the two images, as seen through the eyepiece, were in coincidence. He also described a method for determining curvature by interposing a plate of plane glass between the curved mirror and a source of light.

Mr. ACKERMANN exhibited two experiments: (1) The blowing-out of a candle-flame by the air from a deflating soap-bubble. The bubble was blown at the mouth of an inverted beaker by breathing into a hole cut out at the top. This hole was then presented to the flame, and the flame was immediately quenched. But if the bubble was blown from ordinary air, with bellows, the flame was merely deflected without being extinguished. (2) It was shown that a miniature boat, provided with a false stern, consisting of a linen diaphragm, could be propelled by filling the hollow stern-space with ether, or with some liquid similarly miscible with water. The motion is due to the continuous release of surface-tension behind the boat.

Prof. Boys said that when he tried, some years ago, to blow out a candle with a soap-bubble filled with common air, he found the operation very difficult,—so difficult that, having once succeeded, he never repeated the attempt. It had not occurred to him, as it had to Mr. Ackermann, that the CO_2 present in the breath played a part in the quenching. With regard to the second experiment, he had seen a small boat propelled by dissolving camphor astern, but he thought the use of a liquid for that purpose was a novelty.

The PRESIDENT proposed votes of thanks, and the meeting was adjourned until November 12th.

NOTICES OF BOOKS.

The Principles of Chemistry. By D. MENDELEEFF. Translated from the Russian (Sixth Edition) by GEORGE KAMENSKY, A.R.S.M., of the Imperial Mint, St. Petersburg; Member of the Russian Physico-Chemical Society. Edited by T. A. LAWSON, B.Sc., Ph.D. In Two Volumes. London, New York, and Bombay: Longmans, Green, and Co. 1897.

It is not surprising that a new edition of D. Mendeleeff's *opus magnum* has been required and has accordingly appeared.

The introduction, besides other useful and necessary matter, contains correct definitions of a variety of terms occurring in technical and scientific discussions, and at present grossly misused. A signal instance may be found in the word "phenomenon" and its paronyms.

Of the principles or generalisations of chemical science, the Periodic Law is justifiably spoken of as the most important step in the development of chemistry since the establishment of the atomic theory. The author does not discuss the various, and to some degree conflicting, claims of different chemists to the honour of the first or original discovery.

In the introduction it may be questioned whether the phlogistic theory and its leaders Becher and Stahl are not treated with a too benevolent neutrality. Indeed, Professor Mendeleeff is evidently no controversialist; he draws a parallel between Lavoisier and Dalton on the one hand, and Copernicus and Kepler on the other; but he recognises that the molecular world has not yet found

its Newton. He calls attention to the study of Professor Kononoff on compact phenomena.

Concerning the waters of well known rivers, the author points out that the Neva is remarkable for the small amount of solid matter which it contains, *i.e.*, per cubic metre 55 grms., of which 32 grms. are incombustible and 23 grms. organic.

The constitution of salts was a matter of much discussion. The binary theory dates from Rouelle and Lavoisier; the electro-chemical phase of the question was especially upheld by Berzelius; and the hydrogen theory, which is at present dominant, is due to Davy and Liebig.

Ozone has also proved a bone of contention; the question of its presence in atmospheric air is still open. Ilseay de Ilseay concludes, from a prolonged course of experiments, that the phenomena recently ascribed to ozone are probably due to nitrous acid.

In Volume II., after a table of the periodicity of the elements, we find an account of the elements as demonstrating the author's theory. We find the relations of platinum, palladium, and nickel, and again gold, silver, and copper, distinctly brought into view.

The author contends that the periodic law enables us to see a regularity in the variation of all chemical and physical properties of elements and compounds, and has rendered it possible to foretell the properties of elements and compounds as yet uninvestigated by experimental means.

Copper, silver, and gold melt far more easily than platinum, palladium, and nickel, whilst zinc, cadmium, and mercury melt still more easily. Nickel, palladium, and platinum are very slightly volatile; copper, silver, and gold more volatile; whilst zinc, cadmium, and mercury are among the most volatile metals. Zinc is oxidised more readily than copper, and is reduced with more difficulty, and the same holds good for mercury as compared with gold, whilst the properties of cadmium are intermediate in their respective groups.

Attention is briefly called to the want of two elements whose existence is not sufficiently demonstrated—ekacadmium and actinium.

Crystalline boron is very closely analogous to diamond, *i.e.*, crystalline carbon. It has the lustre, the high refractive power of the diamond, with which mineral it also competes in hardness.

We find here the comparative analysis of four soils, among which the famous "black earth" claims the first rank on account of its richness in potassium, phosphoric acid, and nitrogen.

As regards the localities for bauxite, the north-west of Ireland is unfortunately omitted. Graham's characteristic names for the different states of aluminium hydroxide—"hydrogel" and "hydrosol"—are quoted with approval; and the part played by aluminium acetate in the dyer's procedures is explained.

The practical importance of the aluminium alloys, especially that with copper, is duly appreciated. Pure aluminium is now employed only for objects which require hardness in combination with a low specific gravity. Notes are given on the separation of the rare metals as indicated by Sir William Crookes and others. Doubts are entertained as to whether Welsbach's fractionation of didymium into neodymium and praseodymium can be regarded as final. Becquerel resolves didymium into six individual elements and Sir William Crookes has proceeded still further.

The reader is reminded that the researches of Graham point to the transition from inorganic to organic compounds.

The author gives the general average distribution of phosphoric acid in soils and earthy substances in nature as 1 to 10 parts in 10,000 parts. Its presence in excess is no less pernicious to vegetation than its absence. The red variety—commonly, but erroneously, known as amorphous phosphorus—does not appear to be poisonous. There is a further variety known as metallic phosphorus;

t stands nearer to nitrogen than the yellow variety. The spontaneous inflammability of the hydride PH_3 is mentioned as an interesting fact; possibly the formation of a trace of this variety may account for the phenomena of the *Ignis fatuus*.

The difference between the reactions of ortho-, meta-, and pyro-phosphoric acids the author considers of the deepest interest as regards the theory of hydrates and solutions, which has not yet been fathomed.

Though arsenic is closely analogous to phosphorus, it has a certain resemblance and even isomorphism with the corresponding compounds of sulphur.

The arsenical mirror is a special variety of metallic arsenic, whilst the brown product found simultaneously in the Marsh apparatus is AsH_3 .

The whole of this work has the merit of presenting the loftiest generalisation in harmony with, and based upon, a careful study of facts.

Elements of Chemistry. By RUFUS P. WILLIAMS. Pp. 412. Boston, U.S.A., and London: Ginn and Co., The Athenæum Press. 1897.

THE method of teaching adopted in this book is the result of the author's experience with some 2500 pupils; one great point being his endeavour to make the work have a real fascination for the student by awakening in him his sense of enquiry.

What proportion of time should be devoted to laboratory work has long been a matter of discussion. Students differ considerably, but after some experiments on a large scale, Mr. Williams concludes that, with four or five hours a week for chemistry, less than half should be spent in laboratory work.

The book is divided into forty-one chapters, so we cannot take them seriatim; suffice it to say that the whole subject of elementary chemistry is carefully explained and amply illustrated by formulae, equations, and exercises.

The subject of valence, Chapter X., may be noted as being treated in a specially clear manner.

In Chapter XLI. a brief reference is made to microbes and bacteriology, but the subject is naturally too far advanced to be thoroughly treated in a book for beginners.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Moniteur Scientifique,
Series 4, Vol. xi., September, 1897.

Application of Electrolysis to the Manufacture of Inorganic Products.—L. GOURWITSCH.—The applications of electrolysis to inorganic chemistry are not very varied; they consist for the most part in decomposing salts, and the secondary reactions which occur are only very rarely synthetical. The object of this paper is to review the present state of the question, and numerous references are made to the original papers. The principal subjects touched upon are the electrolytic manufacture of alkalis and chlorine, by the electrolysis of melted chlorides, by methods founded on the use of mercury as the cathode, and by the electrolysis of hydrochloric acid. The electrolytic manufacture of chlorates, hypochlorites, persulphates, permanganate of potash, chromates, bichromates, &c., is also described.

Chemical Modifications which take place in Fruits during their Growth.—C. GERBER.—The author finds that the acids in fruits disappear, giving off at the same time more carbonic acid gas than they can borrow from the

atmospheric oxygen; they form hydrates of carbon. The tannins disappear, on the contrary, when the respiratory quotient is below unity. They do not form hydrates of carbon: so long as tannins exist the fruit will not soften. As soon as the tannins have disappeared softening commences; then follows obstruction of the intercellular meatus, alcoholic fermentation, and the formation of perfumed ethers; at the same time the respiratory quotient becomes greater than unity.

Estimation of Sulphuric Acid. Gravimetric and Volumetric Methods.—F. MARBOUTIN.—Will be inserted at length.

On some New Sulphurised Colouring Matters.—R. VIDAL.—In 1893 the author patented a process for the manufacture of black dyes for cotton, resulting from the action of sulphur on hydroquinone in the presence of ammonia. Some time after that the Société des Matières Colorantes, at St. Denis, took out a patent for the same purpose, by the action of sulphur on paramidophenol. This reaction the author claims to depend entirely on his first patent. It is a very general reaction, and can be applied to all the di- and tri-substituted derivatives of benzene and naphthalene which have two amidised functions, or one amidised and one hydroxylised function, situated in para-.

MISCELLANEOUS.

The Commercial Development Corporation, Ltd.—This Company has been formed primarily to purchase, and thereafter work and commercially develop, Mr. J. G. A. Rhodin's patent improved Electrolyser, which, the Prospectus states, is "an improved process for the electrolytic production of alkali and bleaching-powder, which are two of the staple commodities of the world in constant and ever-increasing demand." Patents have already been granted or applied for in Great Britain, the Colonies, the United States, and the various Continental countries. The purchase price for the patents has been fixed at £90,000, payable as to £20,000 in fully-paid shares, £10,000 in deferred shares, and £60,000 in cash. Dr. John Hopkinson, F.R.S., has been retained as Technical Adviser. The Company also proposes to acquire, as occasion arises, other patents, business, and properties. The capital is fixed at £200,000, divided into 190,000 Ordinary Shares of £1 each and 10,000 Deferred Shares of £1 each. The present issue consists of 90,000 Ordinary Shares of £1 each and 10,000 Deferred Shares of £1 each, of which 70,000 Ordinary Shares are offered for subscription; £100,000 being reserved for further issue if and when required. The Prospectus states that "sufficient capital for the present needs of the Company having been subscribed by the Directors and their friends, the Company will at once proceed to allotment on the closing of the lists," the date of which is fixed at Monday next, November 8th, at 4 p.m., for both Town and Country.

Brussels International Exhibition.—The Horsfall Furnace Syndicate have been awarded, at the Brussels International Exhibition, a Gold Medal for their patent Refuse Destructors, and a Bronze Medal for their patent Smoke-consuming Boiler Furnace.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 1st inst., Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, presiding. The following was elected a Member:—Mr. John W. Woodall, J.P. The special thanks of the Members were returned to Dr. A. J. Hipkins for his valuable present of the Collection of Tuning-Forks made by the late Dr. Alexander J. Ellis, F.R.S., M.C.I.

Technical Instruction at Manchester.—In July and August of the present year a Committee of gentlemen, nominated by the Technical Instruction Committee of the city of Manchester, was deputed to visit certain institutions and schools on the Continent devoted mainly to scientific and artistic instruction as applied to commercial and industrial pursuits. Nine towns in Germany and Austria were visited, and the report of the Committee is now issued. The Act of 1889 establishing technical instruction in England and Wales has awakened much interest throughout the country on the subject of industrial scientific teaching and training; it has also directed the attention of the educational authorities of foreign countries to the efforts being made in England, with the consequence that there has been a considerable development, throughout Germany at least, of educational means and resources. To this may be attributed Germany's great commercial and industrial progress of recent years. It was therefore considered to be of the highest importance to see what the Continental countries have done of late; hence the appointment of this committee. That Germany is in a prosperous condition, due to her successful manufacturing and commercial enterprise, is evident in the extension of her cities, the making of new streets, and the erection of handsome buildings which is going on in every town, and it seems certain that it is the technical teaching which is at the root of this surprising development. We sincerely hope that the report of this Committee will arouse many of our large towns from the apathy which they now show, and be the means of further endeavours for the maintenance of our commercial supremacy, once unchallenged, but now so seriously threatened, both by competitors abroad and so-called "labour leaders" and Trades Unions at home.

NOTES AND QUERIES.

Enamelling Cast-Iron Pots.—I should be obliged for information as to the best material and process for enamelling cast-iron pots to withstand 180° Tw., or the names of enamel pot makers.—**ENAMEL.**

MEETINGS FOR THE WEEK.

FRIDAY, 12th.—Physical, 5. "On the Isothermals of Ether," by J. Rose Innes. "On the Variation with Temperature of the Electromotive Force of the H-form of Clark Cells, by F. S. Spiers and F. Twyman.

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Dr. ALEXANDER SCOTT, M.A., D.Sc.

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All persons desiring to be admitted as workers, must send evidence of scientific training, qualification, and previous experience in original research, along with a statement of the nature of the investigation they propose to undertake.

The terms during which the Laboratory is open are the following—**MICHAELMAS TERM**—First Monday in October to Saturday nearest to the 18th of December.

LENT TERM—Monday nearest to the 15th of January to the second Saturday in April.

EASTER TERM—First Monday in May to the fourth Saturday in July.

Candidates must apply for admission during the course of the preceding Term.

Forms of application can be had from the ASSISTANT SECRETARY, Royal Institution, Albemarle Street, W.

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CIVIL SERVICE COMMISSION.

FORTHCOMING EXAMINATION.

Dispenser in H.M. Naval Hospitals at Home and Abroad (20 to 25), 17th December. The date specified is the latest at which applications can be received. They must be made on Forms to be obtained with particulars from the Secretary, Civil Service Commission, London, S.W.

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S. A. BARTLETT, Esq., F.I.S., 34, Castle Street, Liverpool.

ABRIDGED PROSPECTUS.

OBJECTS.

THE COMMERCIAL DEVELOPMENT

CORPORATION LIMITED has been formed among other things, to purchase from Mr. A. R. Harvey, the Vendor, Mr. J. G. A. Rhodin's patent improved Electrolyser, including all Foreign and Colonial Patents and Protections already granted or elsewhere obtainable in respect thereof, to work, license, and otherwise fully exploit and commercially develop the same, and to promote and form subsidiary companies for the purchase of the said patents or of royalties and rights from this Company in Great Britain and the colonies and foreign countries.

Patents for Mr. J. G. A. Rhodin's invention have already been granted or applied for in Great Britain and the Colonies, United States, Germany, Austria, France, Belgium, Spain, Italy, Norway, Russia, Denmark, and Sweden.

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The Company have already erected buildings at Clifton Junction, near Manchester, which will shortly be equipped with plant, to at once develop and work the apparatus on a large scale.

Report of Dr. JOHN HOPKINSON, F.R.S., Past President
of the Institution of Electrical Engineers.

Westminster Chambers, 5, Victoria Street, S.W.,
23rd October, 1897.

The Commercial Development Corporation Ltd.

GENTLEMEN,—I have on three different occasions had Mr. Rhodin's experimental apparatus for production of caustic soda by electrolysis under prolonged observation.

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I have to remark that the electric potential required to effect the decomposition is comparatively low. Both facts show advantages with regard to economy in this process.

I am of opinion that the success proved by the small apparatus is such as to warrant an expenditure sufficient to prove Mr. Rhodin's system on the scale you are establishing at Clifton Junction.

I am, Gentlemen, Yours truly
(Signed), J. HOPKINSON.

Dr. John Hopkinson has been retained as Technical Adviser.

OTHER OBJECTS OF THE COMPANY.

The Company has also been formed and registered in order to acquire as occasion arises, other patents, businesses, and other property, within the limits of its Memorandum of Association, as the Directors may consider desirable.

It is well known that a large and exceedingly profitable business is being carried on by many companies having for their object the purchase and re-sale or flotation of existing businesses as going concerns; and the purchase and exploitation of new ventures. The Directors have already had under consideration the purchase of valuable businesses, some of which would bring to this Company a considerable profit.

PURCHASE PRICE OF THE RHODIN PATENTS.

The purchase price for the Rhodin patents has been fixed at £90,000, payable as to £20,000 in fully-paid shares, £10,000 in deferred shares, and £60,000 in cash, leaving £10,000 for working capital out of the £100,000 subscribed.

GENERAL.

There will remain £100,000 for further issue as and when required. This the Directors consider ample for all purposes of the Company.

The necessary capital for promoting the Company and working it has already been subscribed or guaranteed by the Directors and their friends; and the Directors have every confidence in the success of the Company. They are all men of high professional or commercial standing.

All charges and expenses (other than registration fees, duties, stamps, and brokerage fees) in connection with the formation of the Company up to date of allotment will be paid by the Vendor.

The following contract has been entered into:—7th October, 1897. An agreement of this date made between Alfred Robert Harvey of the one part, and Samuel Abraham Bartlett, as a Trustee for the Company, of the other part.

The Contract, the Memorandum and Articles of Association, and copies of the Reports, including those upon which the estimates of profits are based, may be seen by intending subscribers at the offices of the Company's solicitors. Other agreements and arrangements have been or may be entered into as to the formation of the Company, and the guarantee and issue of the necessary capital, to none of which the Company is a party.

As these contracts and arrangements may technically be termed contracts within the meaning of the 38th Section of the Companies' Act, 1867, or contracts to the disclosure of which applicants for shares may be held to be entitled, applicants for shares are to be deemed to have agreed with the Company (as Trustees for the Directors and other persons liable) to waive all claims (if any) against them for not more fully complying with the requirements of the said section, or under the Directors' Liability Act, 1890, in respect of any statement in the prospectus made by the Directors or other persons liable in the belief that it was true, and applications will only be received on this express condition.

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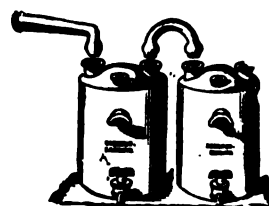
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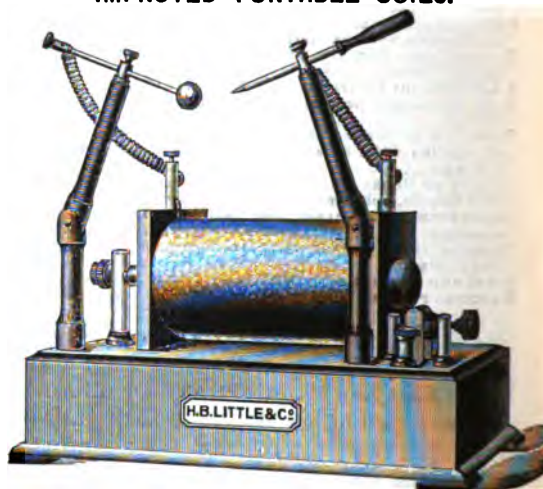
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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1981.

THE SPECTROGRAPHIC ANALYSIS OF MINERALS AND METEORITES.*

By Professor W. N. HARTLEY, F.R.S., and HUGH RAMAGE.

In the course of a spectrographic study of the basic Bessemer flame at Middlesborough the element gallium was found in the iron and was traced to the Cleveland clay ironstone. The process employed in the analysis of the ore was partly chemical and partly spectrographic, and other samples of ores were examined in a similar way (*Proc. Roy. Soc.*, lx., p. 393). The results were satisfactory as far as the detection of gallium was concerned, but the process occupied too much time. The same ores were then examined by a simpler process, in which 0.5 grm. of the powders were rolled in filter-paper and heated in the oxyhydrogen flame, the spectrum of the flame being meanwhile photographed. This operation lasted only about three minutes, and the test for gallium was almost as sensitive as the combined chemical and spectrographic method performed on a much larger quantity, and, in addition, it revealed the presence of many other elements.

A large number of minerals, commercial products, meteoric irons, and meteorites have been examined by this process and very interesting results have been obtained (*Journ. Chem. Soc.*, 1897, pp. 533 and 547). The spectrograph used in this work was described by the aid of a photograph, and spectrograms taken with the instrument were projected directly on the screen.

The simple character of the oxyhydrogen flame spectra as compared with spark spectra was illustrated by examples of the spectra of iron. The former spectra are such that, with the aid of a superimposed spark spectrum of two alloys which give easily recognised lines, the elements may be identified without scale or micrometer measurement. In a mineral or complex mixture of substances the presence or absence of about one-third of the elements may in this way be decided upon in a few moments.

Spectrograms of the following elements were projected on the screen and the lines by which they are most easily recognised indicated:—

Lithium, sodium, potassium, rubidium, and caesium.
Copper, silver, and gold.
Calcium, strontium, and barium.
Zinc and cadmium.
Gallium.
Manganese.
Iron, cobalt, and nickel.

The spectrograms of a number of minerals and meteoric irons were also exhibited, special attention being drawn to the lines of the more interesting constituents.

Tabulated results of the analyses of 169 minerals were given, as in *Engineering*, lxiv., p. 395, and of 13 meteorites, as in a paper read before the Royal Dublin Society in May last, but not yet published in the *Proceedings*.

The Sanitary Institute.—A Sessional Meeting of the Institute will be held at the Parkes Museum, Margaret St., W., on Wednesday, November 19th, at 8 p.m., when a Discussion will take place on "The Pollution of Water Supplies by Encampments of Hop-Pickers, Casual Workers, Tramps, &c." To be opened by Prof. W. H. Corfield, M.A., M.D. (Oxon), F.R.C.P., in reference to the Dangers of Pollution of Municipal Water Supplies; and by Miss M. A. Chreiman, in reference to the Sanitary Control of Hop-Pickers, &c. The chair will be taken by Sir Douglas Galton, K.C.B., D.C.L., LL.D., F.R.S.

* Abstract of a Paper read before the British Association (Section B), Toronto Meeting, 1897.

THE INTERACTION OF HYDROGEN SULPHIDE AND COPPER SALTS.

By JOHN B. COPPOCK, F.C.S.

In the *CHEMICAL NEWS* (vol. lxxiii., No. 1906) I gave an account of some results obtained in connection with the interaction of hydrogen sulphide and copper sulphate,—investigations which took their origin in the statement that cupric sulphide (CuS) was not produced in the interaction, but Cu_4S_9 .

Dr. Brauner in an article (vol. lxxiv., p. 99) pointed out that the precipitates he obtained in working upon this subject contained varying proportions of copper and sulphur, that their composition was not always Cu_4S_9 , and they never contained copper and sulphur in the atomic proportion CuS , except when the original precipitate was left unwashed with carbon bisulphide. Then the precipitate contained copper and sulphur "in the exact atomic proportions $\text{Cu} : \text{S} = 1 : 1$," but some of the sulphur existed in the free state.

Thus it appeared as a point worth noticing that a substance in the process of formation should exert an influence in forming a mechanical mixture of itself and the uncombined negative radicle in exact atomic proportions. This may be but a coincidence, but it suggests the idea that when a lower sulphide is being produced, if free sulphur is formed at the same time, then the lower sulphide and the free sulphur are in the exact atomic proportions required by the higher sulphide.

Analyses of these unwashed precipitates led me to the conclusion that the free sulphur mixed with the copper sulphide precipitate was a very variable quantity, which depended very much upon the time the current of gas was passing through the copper salt solution.

There can be no doubt that the precipitate yielded by the interaction of copper salts and hydrogen sulphide do not always agree with the formula CuS .

Dr. Brauner points out that these precipitates can be represented dualistically as a mixture of Cu_2S and CuS . Several of the precipitates obtained by me, deviating from the formula CuS , I found, after reading Linder and Picton's work (*Chemical Society's Transactions*, 1892), could be represented as compounds of their suggested type. They showed that compounds of the form $n\text{CuS}, \text{H}_2\text{S}$ were formed by interaction of the two above specified substances, establishing the existence of—



What interpretation is to be put upon the fact that the precipitate yielded by the interaction of hydrogen sulphide and copper salts—omitting free sulphur from consideration, as undoubtedly this may be removed by washing—does not always agree with the formula CuS .

Dr. Brauner argues in favour of the production of mixtures of Cu_2S and CuS , which he says finds support, in the action of hydrogen sulphide upon solutions of antimonious and arsenious salts, where mixtures of the lower and higher sulphides are formed, and the tendency which copper has of forming sulpho-salts, as is the case with arsenic and antimony.

That copper does form sulpho-salts—double sulphides—is a fact, but whether it forms a basis for any analogy with arsenic and antimony is doubtful. From considerations of relationship one would expect the production of the compounds $n\text{CuS}, \text{H}_2\text{S}$ to be most likely, as demonstrated by Linder and Picton's work. If hydrogen finds its proper place at head of Group I. (Periodic Law), we should expect it to resemble the other high members of this group, and give combinations with cupric sulphide, as in the cases of sodium and potassium sulphides. Double sulphides are thus known, but they are not formed with the ease and direct manner as are the double sulphides of antimony and arsenic, whose sulphides are very soluble in alkali sulphides, but cupric sulphide is relatively insoluble. The very little analogy between the

two cases does not warrant the idea of an exact resemblance in the interaction of antimonious and arsenic salts with hydrogen sulphide and copper salts with hydrogen sulphide. Moreover, the sulphides of nickel and mercury show a slight solubility in alkali sulphides like copper.

The following method gave almost uniform results in testing whether cupric sulphide could be prepared by action of hydrogen sulphide upon copper salt solutions:—

An unweighed quantity of copper sulphate was dissolved in water, acidified with nitric acid. Nitric acid was added on the idea that any reducing action hydrogen sulphide may possess would be spent upon the acid rather than upon the copper sulphate, and so preventing the production of cuprous sulphide.

The copper sulphate solution was run gradually into hydrogen sulphide solution. The precipitate, after collection, was washed in a cylinder by agitation with dilute acid, then washed free from acid, then left in contact with carbon bisulphide for days, seven at least, during which shaking up went on at times; finally it was washed with alcohol. The precipitate was of a very dark olive-green colour. It is needless to say during these operations oxidation was reduced to a minimum, and apparently none went on.

The precipitate was then dried in an atmosphere of carbon dioxide at a temperature never higher than 100°, inasmuch as it has been shown cupric sulphide reduces to cuprous sulphide when heated to 130°. The precipitate was then fractionated upon the idea that if there were Cu_2S or free sulphur in it such might not be uniformly diffused. Weighed portions were taken and determined by conversion into sulphate and precipitation as barium sulphate, and other portions were digested with strong hydrochloric added, got into solution, oxidised, and the copper determined as cupric oxide. In the oxidation to barium sulphate a little sodium chloride was added, to convert any free sulphuric acid into sodium sulphate, inasmuch as free sulphur was liberated. The following results, and others, were got:—

CuS taken.	CuO found.	Theory for CuS.
0.33	0.2741	0.2744
0.56	0.4652	0.4654
CuS taken.	BaSO_4 found.	Theory for CuS.
0.276	0.6775	0.6770
0.432	1.0616	1.0596

We may therefore conclude that cupric sulphide may be prepared by this interaction. Linder and Picton, in 1892, concluded that in the presence of acid the molecule $(\text{CuS})_n$ was formed as a final breaking-up product of compound between it and hydrogen sulphide.

These investigations have suggested the possible power that precipitates, particularly flocculent ones, may have of retaining finely divided sulphur and hydrogen sulphide, physically or chemically, in their pores, and experiments are contemplated under these headings with other substances than sulphides.

Chemical Laboratory,
Harris Institute, Preston.

ON THE ESTIMATION OF SULPHURIC ACID. GRAVIMETRIC AND VOLUMETRIC METHODS.*

By FELIX MARBOUTIN.

THE large number of volumetric methods for the estimation of sulphuric acid contrasts strangely with the one gravimetric method used. We propose to give a short résumé of the more frequently used volumetric methods, classifying the various processes; we will then describe

in detail a volumetric method which has given us excellent results in the analysis of water. We also think it advisable to describe in some detail the gravimetric method we employed, as it has served as a check on our volumetric method.

I. Gravimetric Analysis.—A volume of water, sufficient to contain 50 to 100 m.grms. of sulphuric anhydride, is concentrated to about 100 c.c. after the addition of a few drops of hydrochloric acid. It is kept at a temperature near boiling, without, however, quite reaching that point; we then add, drop by drop, 10 c.c. of a solution of chloride of barium, of 175 grms. of the crystalline salt per litre. This done, we leave it in a warm place (40°) for twelve hours, wash by decantation with warm water, until nitrate of silver no longer gives a precipitate in the washings; throw on a filter, and again wash with warm water, then place the funnel in an oven, and leave it at 110° for three hours; then put the filter and its contents in a platinum crucible, the point of the filter upwards, and calcine strongly in an oxidising flame; there is then no risk of reducing the sulphate of barium.

This method gives perfect satisfaction; the precipitate is granular, and never passes through the filter, and the use of sulphuric or nitric acid to re-convert the sulphide of barium to sulphate is rendered unnecessary, an appreciable advantage when there are many estimations to make.

In certain cases (highly coloured waters) it is as well to make sure that the precipitate does not contain any foreign matters; for this purpose, after weighing, we fuse with carbonate of soda, then dissolve in water. The solution, acidulated with hydrochloric acid, is precipitated as before.

Ferrous or ferric salts might give a precipitate containing iron, by the formation of a hydrated ferric-barytic sulphate; it is therefore advisable to get rid of these salts if they are present in too great quantity.

When concentrating large volumes of water it is as well to use a vacuum, for the sulphurous products from the gas flame may easily cause the formation and absorption of 10 to 14 m.grms. of sulphuric acid in six hours if the vessel is uncovered. In the case of sewage waters, &c., it is necessary to get rid of the organic matters by evaporating with fuming nitric acid, or by treating with bromine; but care should be taken not to use an excess or to carry the evaporation too far.

2. Volumetric Analysis.—Many methods have been proposed; some specially for alkaline sulphates, others for cases when no chlorides are present, others again are almost universal, but all require several titrated solutions, which causes a considerable amount of uncertainty. Our method, which we shall describe after reviewing those more generally known, presents, we think, certain advantages over those in common use.

I. Direct Method by Chloride of Barium.

A. Houzeau in France, and Wildenstein in Germany, proposed precipitating the sulphuric acid by a titrated solution of chloride of barium, the end of the operation being determined by the absence of precipitation. The determination point, however, is very difficult to decide, and it must not be forgotten that there is a neutral point, where—though there is still some sulphuric acid present—no precipitate is formed unless excess of chloride of barium be added.

II. Direct Alkalimetric Methods.

1. Using Salts of Barium.—F. Mohr added an excess of carbonate of barium in the presence of a current of carbonic acid; the alkaline sulphates form sulphate of barium, and the alkaline carbonates formed are estimated by means of a titrated acid solution. Böhlig has improved this method by working at the boiling-point, but it is not applicable except to the sulphates of the alkaline earths.

2. Using Salts of Strontium.—Rose showed the com-

* Abridged from the *Moniteur Scientifique*, Series 4, vol. xi., September, 1897.

plete transformation of sulphate of strontium into carbonate, by digesting with alkaline carbonates. Mohr and Classen devised a method founded on this principle. The sample was acidulated with HCl, treated with chloride of strontium, and an equal volume of alcohol added. The washed precipitate is digested with carbonate of soda, and dissolved in excess of titrated HCl. The excess is measured with a titrated potash solution.

3. *Using Salts of Lead.*—The sulphate of lead obtained by precipitating with the nitrate is washed, transformed into carbonate by digesting with carbonate of ammonium, dissolved in excess of titrated nitric acid, and the excess of acid measured with titrated soda. This method cannot be used in the presence of chlorides.

4. *Using Caustic Baryta.*—J. Grossmann precipitates the sulphuric acid by caustic baryta; the excess of hydrate of baryta is treated with a current of carbonic acid, the excess of which is driven off by boiling. The soda is titrated alkalimetrically.

5. *Transforming the Precipitated Sulphate into Sulphide.*—According to Linossier the acid may be separated from the solution as an insoluble compound and converted into sulphide. This latter is titrated alkalimetrically with a normal soda solution, using Poirrier orange as indicator. In the case of sulphuric acid it is precipitated by acetate of lead, and changed into sulphide with sulphuretted hydrogen.

III. Indirect Alkalimetric Methods.

1. C. Mohr precipitates the sulphuric acid by an excess of chloride of barium, in a neutral or very faintly acid solution. The excess of chloride of barium is treated with carbonate of ammonia or soda, the precipitate is washed thoroughly, and the carbonate, dissolved in an excess of titrated acid, is determined by means of a titrated alkaline solution. Ad. Clemm has made this method more practicable by adding, after precipitating with chloride of barium, a quantity of carbonate of soda capable of precipitating all the chloride of barium used. The excess of carbonate of soda remaining measures the quantity of sulphuric acid present.

2. Böhlig precipitates with carbonate of baryta in presence of a current of carbonic acid; the alkaline sulphates are transformed into bicarbonates, and the sulphuric acid is precipitated. The carbonates obtained by boiling are estimated with titrated acid in excess, and titrating back with an alkaline solution.

3. Knöfler makes the sample neutral with titrated hydrochloric acid, then boils to drive off the carbonic acid, and makes the solution neutral to phenolphthalein by means of a titrated solution of carbonate of soda. The sulphuric acid is precipitated with titrated chloride of barium, the phthalein showing when the precipitation is complete; he then adds a slight excess of chloride of barium, which is estimated. Altogether this method requires four titrations, each liable to error, and we do not think it can be employed advantageously in most cases. It is worth noting that readings with phenolphthalein in the presence of carbonates should be made in boiling solutions.

IV. Methods by Precipitation with Chloride of Barium, and Measurement of the Excess.

1. Wildenstein proposed to neutralise the solution from which the sulphate had been precipitated, and to measure the excess of chloride of barium by means of chromate of potassium, the end of the operation being shown by the yellow colour of the liquid; the results, however, are never exact.

2. Precht, using an acid solution, transforms the chromate into bichromate, which he treats with soda to change the colour from red to yellow; the excess of chromate is then measured with a salt of iron, by the touch test. This latter operation allows of the use of cloudy solutions.

3. A. Fellet precipitates the sulphuric acid with an

excess of chloride of barium; the excess is then precipitated by a quantity of potassic chromate equivalent to the total quantity of chloride of barium used. The chromic acid remaining in the solution is thus proportionate to the quantity of sulphuric acid in the sample. The chromic acid is estimated by means of ferrous chloride and titrated permanganate of potash.

M. Quantin claims priority for the above method. His process is very little different, but he considers it necessary to have solutions of chromate and chloride which are in absolute agreement, and he even gives corrections to use in case the chromate is the stronger.

4. Mohr and Classen have described a process based on the same principle, but the chromic acid is titrated with iodide of potassium and hyposulphite of soda, using starch as an indicator; this process cannot be recommended, as it requires too many reagents.

5. Windisch proposed, for estimating the sulphates in brewery waters, a method of estimation very similar to the one we have just described, but the excess of chromic acid is titrated by means of arsenious acid and iodine. The disadvantage of this process is, that it is almost as long as the gravimetric method.

V. Methods based on Precipitation in the form of Sulphate of Lead.

1. Levol was the first to propose the use of salts of lead. He poured a titrated solution of lead into the solution containing the sulphates: the end of the operation is shown by the colour assumed by the iodide of potassium which is used as an indicator.

2. A. Guyard, using the same reagents, puts the solution to be titrated into the burette, and runs off the quantity necessary to decolourise a known volume of titrated acetate of lead, coloured with a drop of iodide of potassium. Again, we can precipitate the sulphates with an excess of acetate of lead, and measure the excess with bichromate and salts of silver. None of the methods based on the action of salts of lead can be applied in the presence of chlorides; this is their weak point, for there are many analyses made where we do not detect the chlorine in the state of combination in which it exists in the sample, or where its presence is due to the preliminary solution of the sample.

VI. Method of Félix Marboutin.

We precipitate the sulphuric acid with chloride of barium; the excess of baryta is then precipitated by a quantity of chromate greater than that required by all the chloride of barium used; the chromic acid remaining in the solution is oxidised by a known volume of an arsenious solution; the excess of arsenious acid is measured with titrated iodine.

The reading of iodine obtained in an analysis of water, subtracted from the reading obtained with distilled water free from sulphuric acid, gives the volume of iodine solution equivalent to the sulphuric acid in the sample.

The following is our method of working:—

One hundred c.c. of the water to be analysed are acidulated with HCl, then boiled to drive off carbonic acid; the temperature is then lowered to just below boiling, and 30 c.c. of chloride of barium are added drop by drop; it is then allowed to stand twelve hours at 40°, for the precipitate to become well agglomerated. Neutralise with a few drops of ammonia, and add 30 c.c. of chromate of potassium.

The liquid is gently heated, and then after cooling made up to 300 c.c. 100 c.c. of the clear liquid is taken and 2 c.c. of $\frac{1}{2}$ sulphuric acid, and 5 c.c. of arsenious acid are added; it is then gently warmed and shaken until completely decolourised. To the solution, neutralised with carbonate of potassium, we add from a burette a titrated solution of iodine, with starch as an indicator.

t = the value of 1 c.c. of iodine in m.grms. of I.

n = the number of c.c. of iodine used with 100 c.c. of distilled water.

n_1 = the number of c.c. of iodine used with 100 c.c. of the sample.

x = the number of m.grms. of sulphuric anhydride in the sample.

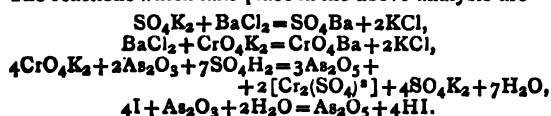
Then—

$$x = 10 (n - n_1) 3 \frac{4 \times 80}{3 \times 4 \times 127} \cdot \frac{N}{50}$$

If the solution of iodine is exactly $\frac{N}{50}$, that is to say, if 1 c.c. of iodine is equal to 2.54 m.grms. of iodine,

$$x = 16 (n - n_1).$$

The reactions which take place in the above analysis are—



The solutions used are—

Crystallised chloride of barium, 4.8 grms. per litre, $\frac{N}{50}$,

Crystallised chromate of potassium, 3.9 " $\frac{N}{50}$,

Arsenious solution, 4.95 grms. arsenious acid per litre.

Dissolve 4.95 grms. of arsenious acid in water containing 10 grms. of potash, heat gently; after cooling make acid with sulphuric acid, and make up to a litre. Iodide solution: weigh out 2.54 grms. of bi-sublimed iodine, dissolve in water with 5 grms. of iodide of potassium; make up to a litre and titrate with a known weight of hyposulphite of soda.

Our method has several advantages over the others we have mentioned; it has a distinct determination; it does not require filtrations or washings; only one titrated solution is necessary; it takes cognisance of the possible impurities in the reagents used, by a comparative reading with distilled water. It gives results absolutely comparable with those obtained gravimetrically, and that with a considerable saving of time—of at least three-quarters.

It differs from the method described by Windisch, and other methods based on the use of solutions of chloride of barium and chromate of potassium, in that it is not necessary to use a volume of chromate exactly equivalent to the volume of chloride of barium used. We measure, in fact, the difference between the readings made with a water deprived of sulphuric acid and a water containing some. The absolute value of these readings is therefore a matter of indifference.

Our method can be applied to all estimations where sulphuric acid can be isolated by precipitation with chloride of barium. We must, however, point out that in the case of waters containing much organic matter, such as sewage, it is first of all necessary to get rid of this matter, the reading of iodine being in such cases too high, and varying with the nature of the organic matter present. In the case of water from springs, rivers, wells, and surface drainage, the method gives excellent results.

M. Marcel Molimé has compared the figures obtained by our method with those obtained by Windisch's. The difficulty of obtaining solutions of barium, and of chromate, strictly equivalent in the latter case, has led him to determine the corrections necessary from the fact of using solutions not absolutely equivalent.

T_1 being the value of arsenious acid in iodine.

T_2 " " chromate in arsenious acid.

I the reading of iodine in the analysis.

The weight of sulphuric anhydride in Windisch's method is given by—

$$I_2 \frac{T_1 - I}{T_1 \times T_2}$$

We have found that—

1. Five c.c. of arsenious acid decolourises 25.2 c.c. of iodide solution, from which we get $T_1 = 25.2$.
2. Ten c.c. of chromate added to 100 c.c. of water and 10 c.c. of arsenious acid, required 20.6 c.c. of iodine. 10 c.c. of chromium = $(25.2 \times 2) - 20.6$ or 29.80 of iodine.

$$T_2 = \frac{5}{2} \cdot \frac{29.8}{25.2} = 2.956.$$

3. If we use a solution of iodide which is not exactly $\frac{N}{50}$, we must correct the readings, so that they do represent a solution of $\frac{N}{50}$; in the present case the coefficient by which the readings must be multiplied is—

$$C = \frac{25.0}{25.2} 0.992.$$

4. From 30 c.c. of chromate + 30 c.c. of baryta, made up to 300 c.c. with distilled water, we take 100 c.c., to which we add 5 c.c. of arsenious acid. The reading shows 24.70 c.c. of iodine. The difference $25.2 - 24.7 = 0.5$ c.c., measures one-third of the value of the difference which exists between the 30 c.c. of chromate and the 30 c.c. of barium. The formula, calling x the correction to be made, is as follows:—

$$\left[(T_1 - I - x) \right] \frac{0.992 \times 12}{\frac{5}{2} \cdot \frac{29.8}{25.2} 25.2}.$$

$$(T_1 - I - x) \frac{0.992}{29.8} \cdot \frac{2}{5} \cdot 12.$$

$$(25.2 - I - x) 15.98.$$

RELATIONS CONNECTING THE THERMAL CONSTANTS OF THE ELEMENTS.

By NOEL DEERR.

EMPIRICAL relations existing between the thermal constants of the elements have been proposed by Pictet, Richards, Crompton, and Deerr. As these relations are all very similar, and in some cases identical, it is here intended to collect together—and to some extent to criticise—the work that has been already done.

The following notation is used in this article:—

T = Melting-point absolute in degrees Centigrade.

L = Latent heat of fusion per unit mass.

S = Mean specific heat between -273° and T .

C = Mean coefficient of expansion between -273° and T .

A = Atomic weight.

V = Atomic volume.

W = Valence.

To the various calculations the quantities S and C are generally those determined between 0° and 100° . The relations it is proposed to discuss are—

1. $T C \sqrt[3]{V} = \text{constant}$. (Pictet).
2. $T C = \text{constant}$. (Deerr, CHEMICAL NEWS, vol. lxxi, p. 303).
3. $\frac{L C}{S} = \text{constant}$. (Deerr, CHEMICAL NEWS, vol. lxxi, p. 303).
4. $\frac{T S}{L} = \text{constant}$. (Deerr, Proc. Chem. Soc., xvii., 6, 95).

In the relations 2, 3, 4, the constant is stated to hold only between such elements as bear a close relationship to each other, and hence generally for members of any one periodic group.

5. $\frac{AT}{LW} = \text{constant.}$ (Crompton, *Trans. C. S.*, 145,

6. $ALC\sqrt[240]{V} = \text{constant.}$ (Richards, *CHEM. NEWS*,
vol. lxxv., p. 278).

7. $\frac{L}{TS} = \frac{1}{2}.$ (Richards, *Journal of the Franklin*
Institute, 1893).

Before proceeding to discuss these relations, the writer would draw attention to the widely variant values given by different observers to much of the data in question; the writer has been at great pains to use the most reliable determinations, and in such cases where he has not been able to discriminate between different values, both are given.

Of the relations given above, those numbered 4, 5, 7 are identical, with differences of detail; and the main idea of 1 and 2 is the same; the relation 3 follows from a combination of 2 with 4, precisely as 6 is obtained by Richards by combining 1 with 7, and putting $S.A.S=6.4$.

In discussing the dependency of the latent heat of fusion on the total heat at the melting-point, Crompton, who does not introduce the specific heat, but uses by preference the atomic weight, attempts to reduce the values he obtains to one constant by the introduction of the valency. The introduction of this quantity into the equation is, in the opinion of the writer, a disadvantage; for, besides destroying the dimensions of the equation and making the law almost unintelligible, the values so obtained are not constant, varying quite regularly from 0.972 for lead to 1.789 for silver, with a mean value of 1.370; and, further, in the case of bromine and iodine, Crompton has to assume, on very slender grounds, a hypothetical triadic valency, whilst in the case of phosphorus and sulphur he has recourse to the quantity "reciprocal of the valency." Richards, in discussing the relation, gives it a definite physical meaning, stating the latent heat of fusion to bear a constant ratio ($\frac{1}{2}$) to the total heat at the melting-point, but he does not appear to realise the bearing of chemical relationships upon the value of the ratio. Deerr expressed the relation in terms of temperature, naming the quantity $\frac{L}{S}$ the "temperature equivalent of the latent heat of fusion," and he restricted the law as only holding between elements of the same valency.

In Table I. are given values of T, TS, L, and of the ratio $\frac{TS}{L}$.

TABLE I.

Element.	T.	TS.	L.	$\frac{TS}{L}$.
Sodium	365	108.6	32.7	3.27
Potassium	335	56.6	15.7	3.67
Copper	1470	140	43.0	3.25
Silver	1220	69.5	21.1	3.29
{ Silver	1220	69.5	24.7	2.81
Thallium	560	17.4	5.1	3.41
{ Zinc	695	66.0	28.1	2.35
{ Zinc	695	66.0	22.6	2.92
Cadmium	601	34.2	13.6	2.51
Aluminium	1150	256	100	2.56
Mercury	233	7.2	2.8	2.57
Palladium	1700	98.7	36.3	2.72
Gold	1330	42.7	16.3	2.62
Platinum	2300	71.3	27.1	2.60
Gallium	286	21.9	19.2	1.14
Bismuth	535	14.4	12.5	1.15
Lead	693	18.7	5.4	3.47
Tin	508	28.2	14.5	1.90
Bromine	266	27.5	16.2	1.76
Iodine	386	20.8	11.7	1.78
Sulphur	388	69.1	11.8	5.19
Phosphorus	317	59.9	5.0	12.0

Referring to the table, it is seen that the elements Na, K, Cu, Ag, Tl give a constant well within the limits of experimental error. Of these, the first four all belong to the same periodic family; and Tl, although a member of a different family in the thallous compounds, is very similar to the alkaline metals. Following these, there are seven elements giving a value closely approximating to 2.5. Of these, Zn, Cd, Hg are members of one family; Pt, Pd, Au are elements closely allied, although they cannot be strictly said to belong to one family; Al, too, gives a value the same as for these elements, but the data of this element is so uncertain as to render discussion futile. The closeness of the values for the zinc elements and the platinum elements is, in the writer's opinion, accidental, and does not point to an absolute identity. Ga and Bi, although not in the same family, give identical values. These elements are, however, allied; both being trivalent and presenting many analogies. The values given by Br and I require no comment. Following on what has been written above, an identity in the values for Pb and Sn would be expected; the discrepancy is not, however, so startling when the marked non-metallic nature of Sn is remembered. The values for P and S, compared with others, appear abnormal. The specific heat of these elements is obtained near to their melting-point, at which it is at its greatest value; as this quantity varies largely, the cause of the high values may lie therein.

The discussion of this relation can be best concluded by using the law to predict unknown latent heats of fusion. In doing so, the writer was at times in doubt in deciding which of the various values to use; in the table subjoined in such cases the writer has given both values. In the case of In he thinks preference should be given to the value obtained from the constant for Na, &c.; the analogy between In and Tl being so marked. And in the case of arsenic he prefers the value obtained by a comparison with phosphorus.

TABLE II.

Element.	T.	L (by T.S. calculation).		Constant used.
Lithium ..	450	41.0	12.4	3.3 Mean of Na, &c.
Magnesium ..	1020	275	106	2.6 Mean of Zn, &c.
Indium ..	450	23.2	7.0	3.3 Mean of Na, &c.
{ Indium ..	450	23.2	20.4	1.14 Mean of Ga and Bi.
Antimony ..	700	37.1	32	1.14 Mean of Ga and Bi.
{ Arsenic ..	685	57.2	50	1.14 Mean of Ga and Bi.
{ Arsenic ..	685	57.2	4.8	12.0 Value for P.
Cobalt ..	1700	182	68	2.65 Mean of Pt, &c.
Iron ..	1800	198	75	2.65 " "
Nickel ..	1900	215	81	2.65 " "
Rhodium ..	2000	122	46	2.65 " "
Ruthenium ..	2300	143	53	2.65 " "
Osmium ..	3000	94	35	2.65 " "
Iridium ..	2800	90	33	2.65 " "
Selenium ..	490	41.2	8	5.2 Value for S.
Tellurium ..	653	30.9	6	5.2 " "
Chlorine ..	200	36	20	1.77 Mean of Br and I.

The discussion of the relations $TC\sqrt[3]{V} = \text{constant}$ and of $TC = \text{constant}$ is rendered difficult by reason of various observers obtaining different values for both T and C. In calculating Table III. the writer has not given the different values obtained, but has used those values which he has considered most reliable. In considering the relation, then, a fairly wide margin must be allowed for experimental error. The writer is so placed as not to have access to Picket's paper, and doubtless some of the values of the thermal constants used by him differ slightly from those used by the writer. In Table III. the values of T are those already given in Table I.

On reference to the values of $TC\sqrt[3]{V}$ it is seen that

TABLE III.

Element.	C × 10,000.	T C.	T C √V.	$\frac{LC}{S}$.	A L C √V.
Sodium	0'71	0'026	0'074	0'0078	0'151
Potassium	0'84	0'028	0'099	0'0079	0'175
Silver	0'20	0'024	0'052	0'0074—0'0086	0'099—0'116
Copper	0'18	0'027	0'052	0'0081	0'094
Magnesium	0'27	0'028	0'077	—	—
Zinc	0'28	0'019	0'040	0'0067—0'0081	0'086—0'107
Cadmium	0'32	0'019	0'044	0'0081	0'113
Mercury	0'61	0'014	0'034	0'0054	0'081
Aluminium	0'23	0'027	0'059	0'0094	0'136
Thallium	0'31	0'017	0'044	0'0050	0'096
Indium	0'42	0'019	0'047	—	—
Lead	0'27	0'016	0'042	0'0047	0'077
Tin	0'28	0'014	0'035	0'0071	0'120
Phosphorus	0'12	0'0039	0'009	0'0003	0'005
Arsenic	0'056	0'0038	0'009	—	—
Antimony	0'11	0'0077	0'020	—	—
Bismuth	0'14	0'0075	0'021	0'0061	0'102
Sulphur	0'64	0'025	0'063	0'0037	0'060
Selenium	0'37	0'022	0'055	—	—
Tellurium	0'17	0'013	0'036	—	—
Iron	0'11	0'020	0'039	—	—
Cobalt	0'13	0'022	0'041	—	—
Nickel	0'12	0'023	0'043	—	—
Rhodium	0'096	0'019	0'039	—	—
Ruthenium	0'085	0'019	0'040	—	—
Palladium	0'11	0'019	0'039	0'0069	0'094
Osmium	0'066	0'020	0'042	—	—
Iridium	0'070	0'020	0'042	—	—
Platinum	0'088	0'020	0'042	0'0077	0'099
Gold	0'145	0'019	0'041	0'0074	0'102
Bromine	3'5	0'093	0'278	0'053	1'39
Iodine	2'35	0'090	0'268	0'051	1'13

twelve elements give values lying between 0'04 and 0'05, that ten give values lying between 0'03 and 0'06, and that an absolute disagreement is presented by the remaining ten. Now, if the values of $T C \sqrt[3]{V}$ are constant in preference to values of T C, the introduction of the quantity $\sqrt[3]{V}$ should at least tend to correct the values of T C; but it is this quantity which makes the value of $T C \sqrt[3]{V}$ so high for Na and K, and in the other discordant cases its introduction has no effect. Then, again, however much the atomic volume varies, its cube root shows but little variation, and the introduction of this quantity has but seldom a deciding effect, and when it does happen to do so its introduction is hostile to the equation; to say the least, the reaction cannot hold in the case of Na, K, Mg, P, As, Sb, Bi, S, Br, I, and for the remaining elements the agreement demanded by the equation is by no means perfect.

When, however, the relation is put in the form $T C = \text{constant}$, and restricted as holding only between related elements, regularities at once appear; between Na, K, Cu, Ag; between In and Cd; between Br and I; between P and As; between Sb and Bi; and between the elements in the short periods of four in the periodic classification the agreement is all that could be desired. A partial agreement is shown by Ti and In, and by S and Se. The relation fails to hold between Pb and Sn, and also fails in the case of Mg and Hg when compared with Zn and Cd, and in the case of Te compared with S and Se.

A point which is worthy of notice in comparing the relations $T C = \text{constant}$ and $T C \sqrt[3]{V} = \text{constant}$ is that the former has a definite physical meaning which is not possessed by the latter. Expressed in words, this relation signifies that the expansion from -273° to the melting-point is constant for related elements, and so is a function of the atomic weight.

There remain for discussion the relations—

$$\frac{LC}{S} = \text{constant} \quad \text{and} \quad A L C \sqrt[3]{V} = \text{constant}.$$

The dependency of latent heat of fusion upon total heat at the melting-point in connection with chemical relationships has already been shown. It follows, then, that this relation must also hold. In this relation the effect of chemical relationships is not so well shown; most of the values obtained approximating to 0'0075. The rejection of the relation $T C \sqrt[3]{V} = \text{constant}$ implies the rejection of $A L C \sqrt[3]{V} = \text{constant}$; a conclusion borne out by reference to the table.

The relation $T C = \text{constant}$ is not without a theoretical basis, and was obtained by the writer on an argument like this:—At the absolute zero the sum total of the attractions exercised on and by the particles composing a body is at its greatest value. When, by the influence of a rise of temperature, the body has become liquid, no appreciable error is introduced if the attraction is put equal to zero. Using the same notation as before, the heat supplied is $T S + L$. Assuming that the proportion of heat supplied used in overcoming internal attraction is constant, the equation $T S + L = m a$ results; where m is a constant and a represents the total internal attraction at -273° .

On similar grounds, the heat necessary to cause a body to expand through any fraction of its original length is a measure of the internal attraction. Taking the fraction as unity the equation $\frac{S}{C} = m a$ results. From these is obtained the relation—

$$\left(T + \frac{L}{S}\right)C = \frac{m}{n};$$

but since, for related elements, the latent heat has been shown to be dependent on the total heat at the melting-

point, it follows that $TC = \text{constant}$ and $\frac{L}{S} = \text{constant}$ for related elements.

The paper can be best concluded by using the relation $\frac{L}{S} = \text{constant}$ to predict unknown latent heats of fusion. It appears that those obtained from this equation coincide remarkably with those given in Table II.

TABLE IV.

Element.	L (by calculation).	Constant used.
Magnesium	70'	0'0077 Mean of Zn and Cd.
Indium	6'7	0'0055 Value for Tl.
Antimony	32'	0'0061 " Bi.
Arsenic	4'6	0'0003 " P.
Iron	75'	0'0075 Mean of Pt, Pd, Au.
Cobalt	63'	0'0075 " "
Nickel	66'	0'0075 " "
Rhodium	48'	0'0075 " "
Ruthenium	55'	0'0075 " "
Osmium	35'	0'0075 " "
Iridium	33'	0'0075 " "
Selenium	8'4	0'0037 Value for S.

Windsor Forest, West Coast, Demerara.

A THEORY OF THE AURORA BOREALIS.

By GUSTAV WENDT.

We have here abstracts from and comments on the researches of Halle, Celsius, Hiorten, Canton, and especially Fritz in his work "Das Polarlicht" (1881, Brockhaus).

We may be reminded, as a not unimportant point in establishing the polar light theory, that oxygen is a magnetic—or, more strictly speaking, a paramagnetic—element, that it assumes polarity by the presence of the earth as a magnet permanently present. As Humboldt remarks, "Every atom of oxygen represents a minute magnet, and in the solid or in the liquid state possesses strong magnetic properties." Hence near the magnetic pole the magnetic attraction occasions the descent of paramagnetic matter, especially oxygen or condensed oxygen, and also dust of all kinds, occasionally metallic dust, especially dust of meteoric iron and of nickeliferous nature. If, according to a large series of recent accurate analyses, the air of the mountains and moors of the Scottish highlands generally contain 21 per cent of oxygen, whilst in the large towns, especially in fogs, it sinks to 20·8, and in deep mines to 20·2 per cent, this fact may be explained by the circumstance that, besides the general diffusion, the magnetic attraction is here brought into play. Every large mountain mass possesses, in a larger or smaller degree, the so-called mountain magnetism. The agreeable sensation in lofty yet protected regions is chiefly due to the presence of condensed oxygen, which is continually drawn downwards in consequence of the "mountain magnetism." That the strength of northern lights is directly connected with the activity of the sun must be considered as demonstrated, and is easily explained by the fact that the strength of terrestrial magnetism increases and decreases in proportion to the activity of the sun.

If Fritz demands categorically that a theory of the northern lights should explain why the phenomenon is only manifested by night, he forgets the observations of Humboldt ("Kosmos," iv., 1858, p. 145), that the phenomenon has been observed near the sun on December 3rd, 1827. Humboldt adds, in a note, that the arches of the northern lights were seen near the sun in North America, in Parma, and at London.

Another feature of the northern lights, i. e., the origin of the so-called spersal polar light ray ($\lambda = 5567$) has

been explained by Wüllmer (*Poggend. Annalen*, N. F. 388, p. 619).

Hence the northern lights may be regarded as an electrical phenomenon arising when oxygen and other paramagnetic matter is continuously drawn down from the higher regions of the atmosphere, thus setting up electric currents.—*Naturwissenschaftliche Wochenschrift*.

EARLY AMERICAN CHEMICAL SOCIETIES.*

By Prof. H. CARRINGTON BOLTON.

(Concluded from p. 227).

Columbian Chemical Society of Philadelphia (continued).

Dr. ADAM SEYBERT (d. 1825) was one of the earliest American chemists to make a series of analyses of the air by eudiometric methods. Having made twenty-seven air analyses during a voyage across the Atlantic, he compared the results with others made on land, and drew the conclusion that the sea exerted purifying power over the air; his paper before the American Philosophical Society bore the date 1797.

Benjamin Silliman is a name so familiar to American chemists as to require no eulogium in this place. At the founding of the Chemical Society he was forty years of age, and had held the chair of chemistry in Yale College for ten years. It should be remembered that he did not begin publishing the *American Journal of Science* until 1818.

Dr. John S. Stringham, professor of chemistry in New York (institution not specified); Dr. Jared Troust (whose name should be written Gerard Troost), lecturer on mineralogy in the Academy of Natural Sciences, Philadelphia, and afterwards professor of chemistry, mineralogy, and geology in Nashville University (1828-50); Lawrence Washington, Esq., of Virginia; and Dr. Caspar Wistar, professor of anatomy in the University of Pennsylvania, with his relative Charles Wistar, complete the roll of home members.

The prominence of medical men on this list is evident, and is easily explained. Before the days of schools of science, and before colleges devoted a portion of their curricula to scientific studies, almost the only training in science received by American youth was in the medical schools. The chairs of natural history and of the physical sciences were almost exclusively held by physicians whose education more nearly qualified them for teaching these branches of knowledge than the graduates of the classical courses customary in all colleges.

To elevate the standard of membership in the Columbian Chemical Society, a number of distinguished foreigners were enrolled. France contributed Adet, Berthollet, Chaptal, Deyeux, Abbé Haüy, Bouillon-Lagrange, Gay-Lussac, Monge, Guyton de Morveau, Parmentier, Pelletier, Sequin, Thénard, and Vauquelin. Great Britain was represented by Sir Joseph Banks, John Dalton, Sir Humphry Davy, John Davy, J. A. de Luc, Hatchett, Dr. William Henry, Sir William Herschel, Dr. John Hope, John Murray, William Nicholson, Dr. G. Pearson, Mr. W. H. Pepsy, Dr. Thomas Thomson, Alexander Tilloch, and Dr. William Hyde Wollaston. Spain was represented by Professor Troust of Madrid, and the other countries of Europe had not a single representative. The absence of such eminent names as Richter, Klaproth, Stromeyer, Trommsdorff, and Gehlen, of Germany, as well as of Berzelius, the Swede, presumably indicates that at this early date communications and exchange of courtesies with Germany and Northern Europe was less common than with England and France.

The Columbian Chemical Society of Philadelphia published in 1813 one volume of "Memoirs."† This forms

* Read before the Washington Chemical Society, April 8, 1897. From the *Journal of the American Chemical Society*, August, 1897.
† Copies of the "Memoirs" are found in Philadelphia libraries, and in the private library of the writer.

a book of 227 pages, octavo, and bears the imprint of Isaac Peirce, No. 3, South Fourth Street, Philadelphia. It contains twenty-six essays, by ten writers, on a great variety of topics, original, speculative, and practical.

No less than eight of the papers are from the pen of Dr. Thomas D. Mitchell, and these I proceed to review. Dr. Mitchell's "Remarks on the Phlogistic and Antiphlogistic Systems of Chemistry" opens the volume. In this essay he supports the Lavoisierian theory of combustion, stating that there is "no necessity for the principle of inflammability;" he cites the experiment of Woodhouse, who obtained an inflammable air by heating charcoal with scales of iron, both being free from water, and points out that Cruikshank, of Woolwich, demonstrated that the inflammable gas thus obtained is gaseous oxide of carbon (carbon monoxide), discovered by Priestley in 1799, and combustible although containing no hydrogen. He compares combustion with neutralisation of an acid and base, saying "Inflammation and acidity are effects resulting from the action of relative causes, and not attributable to a single agent or principle."

Dr. Mitchell's second paper, "Remarks on Heat," deals with speculations on latent heat, objecting to this term and to Dr. Black's theories.

In a paper entitled "On Muriatic and Oxy-muriatic Acids," Dr. Mitchell vehemently attacks the views of Sir Humphry Davy as to the non-existence of oxygen in muriatic acid, clinging to the statement of Lavoisier, that all acids contain oxygen. In a section on combustion he remarks "we have incontestable proof that oxygen gas contains light," and he regards combustion as accompanied by the decomposition of oxygen gas.

Dr. Mitchell's fourth paper is of a more practical character, being the "Analysis of Malachite" from Perkioming, Pennsylvania. The result is given as follows:—"120 grains of the green carbonate contained carbonic acid, 30 grains; quartz and siliceous earth, 68 grains; brown oxide of copper, 15 grains; loss in the process, 7 grains."

The specimen was evidently a poor one; no account was taken of the water, and reporting results in percentages was not in vogue.

In some "Remarks on Putrefaction" the same writer discusses the action of antiseptics, and attributes the virtue of nitrate of potash to the increase of cold produced by the muriate of soda.

Dr. Mitchell's "Chemical View of Secretion," and his "Analysis of Professor Coxe's Essay on Combustion and Acidification," are polemical and speculative; in his "Remarks on the Atmosphere" he argues to prove the atmosphere a chemical union of oxygen and nitrogen.

Franklin Bache contributes three essays to the volume. "An Inquiry into What Circumstances Will Warrant us Justly to Reckon any Substance a Principle of a Common Property of Any Set of Bodies," discusses the much disputed question of that day, whether hydrogen as well as oxygen can be an acid-forming principle. His conclusion being, "it may." Bache's second paper is entitled, "An Inquiry Whether Mr. Berthollet was Warranted from Certain Experiments in Framing the Law of Chemical Affinity, that it is directly Proportional to the Quantity of Matter." In this essay the author points out "the probable way in which this great philosopher fell into this great error." In a third paper styled "Thoughts on the expediency of Changing Parts of the Chemical Nomenclature," Mr. Bache proposes the following names: Nitral acid forming nitrotes, nitril acid forming nitrites, nitrous forming nitrites, and nitric acid forming nitrates, for the several acid-forming oxides of nitrogen. Fortunately his influence was insufficient to inflict these names on chemical language.

Dr. John Manners contributed four papers to the *Memoirs*. (1) "Experiments and Observations on the Effect of Light on Vegetables and upon the Physiology of Leaves," which abounds in quotations from Darwin's "Botanic Garden." (2) "Analysis of a Mineral Spring

at the Willow Grove," (fourteen miles from Philadelphia). In this the author was assisted by Dr. Mitchell. They report the action of each testing solution on the water, and conclude that the water contains iron and sulphuretted hydrogen, and show the absence of lime, copper, and carbonic acid." (3) "On the Production of Sulphuretted Hydrogen by the action of Black Sulphuric Acid Diluted with Water on Iron Nails." The acid had been blackened by a piece of cork which had fallen in. (4) "Experiments and Observations on Putrefaction." In this paper Dr. Manners tested the influence of carbonic acid, hydrogen, and other gases on putrefying flesh, and also attempted to collect and analyse the gases generated by the same. He concludes that "putrefaction depends on a destruction of the equilibrium of attractions which exist in the elementary principles of which the animal substance is composed in a healthy state, occasioned by the loss of vitality in consequence of which new compositions and decompositions ensue."

Professor Cutbush, President of the Society, wrote "On the Prognostic Signs of the Weather," and "On the Oxyacetate of Iron as a test for the Discovery of Arsenic;" the latter being a good presentation of his discovery, subsequently used as a quantitative method by Kotschoubey.

Mr. Samuel F. Carl, one of the junior members, has two papers containing analyses, the first of the mineral spring at Bordentown, New Jersey, which proved to be a "carbonated chalybeate water," and the second of two specimens referred to him by the society; these proved to be respectively an iron ore and a ferruginous copper ore. The method of reporting results seems very crude to a modern analytical chemist.

Dr. Joel B. Sutherland contributes "Speculations on Lime," in which he claims that if mortar be made with sand containing common salt, the resultant compound gives so much coldness to the mass that during the whole summer vapour is almost incessantly precipitated on the wall with which it is plastered. He also wrote "A Few Remarks on the Nature of the Nervous Influence." Akin to the latter are the "Thoughts on the Principle of Excitability," by George Ferdinand Lehman, who also wrote "On the Emission of Oxygen by Plants."

Mr. William Hembel, Jr., has two papers, one entitled "Observations on the Formation of Muriate of Potash in the Process of Preparing the Hyperoxymuriate of Potash," which is complicated by the belief that hydrochloric acid is an oxygen compound; and another entitled "A New Method of Mounting Woulfe's Apparatus," which is unintelligible owing to the omission of a woodcut to which the text refers.

Mr. Edward Brux, of France, one of the junior members, writes "Upon the Effects of Various Gases upon the Living Animal Body," which consists largely of speculations: notwithstanding which he cites an admirable passage from Dr. Bostock; "Physiologists have, in general, been more inclined to form hypotheses than to execute experiments, and it has necessarily ensued from this unfortunate propensity that their science has advanced more slowly than perhaps any other department of natural philosophy." Unfortunately this truth was not fully recognised by the members of the Columbian Chemical Society.

A contemporary journal (N. Y. Medical Repository), in reviewing the "Memoirs," uses the following quaint language: "It is highly gratifying to behold a band of worthies like those before us, laboring to analyse the compounds which they find ready made, to form by synthesis new combinations in the laboratory, and thereby to deduce correct doctrines from the facts which are disclosed. We cordially congratulate them on their noble occupation and on the progress they have made. We hope they will be persevering and undaunted. And if from this beginning there shall arise great improvements in theoretical disquisition, as well as in economical exercise, we shall rejoice with a mingled glow of amicable and patriotic sentiment."

III. The Delaware Chemical and Geological Society.

The Delaware Chemical and Geological Society was organized at Delhi, Delaware County, New York, September 6, 1821; the first meeting was held at the hotel of G. H. Edgerton in the village, and the following officers were chosen:

President—Charles A. Foote.
Vice-President—Rev. James P. F. Clark.
Recording Secretary—Charles Hathaway.
Corresponding Secretary—Dr. Calvin Howard.
Treasurer—Selah R. Hobbie.
Directors—Cornelius R. Fitch, R. W. Stockton, Ebenzer Steele.

The society was composed of "between forty and fifty well-informed and respectable inhabitants" of the County of Delaware. The following gentlemen were elected corresponding members: Colonel Henry Leavenworth, U.S.A.; Edwin Crosswell, of Catskill, and O. Rice, of Troy.

The society had for its object the improvement of the members in literature and science, especially in chemistry and mineralogy. The members planned to form a library and to secure a chemical laboratory; they made a collection of the minerals and rocks of the region, which was still preserved in the Delaware Academy in 1856. The meetings of the society were held quarterly, and at each an essay or an "original scientific discourse" was presented; it was, however, not long sustained.

In reviewing the condition of chemical science in the United States, as indicated by the membership and achievements of these early societies, we note that those who held the most prominent places were handicapped by the necessity of devoting a large part of their intellectual energy to topics quite outside of the domain of chemistry itself. The active members were either busy with the art of healing, or with teaching several branches of the physical and natural sciences, and too often chemistry was regarded in the colleges as a kind of side issue, or appendix to the more important subjects of instruction. This was caused by the necessity of earning a competence at a time when there was no opportunity of reaping pecuniary rewards by skill as an analyst, or by the application of science to the manufacture of products involving chemical knowledge. Indeed, in default of this stimulus to laboratory work, it is not surprising that the papers read to the societies were largely either reviews of the grand discoveries made by Europeans, or essays in which the imaginative faculty was given free play, it being far easier to indulge in speculation than to discover new facts.

In the early struggles of a country to secure a place among nations, few men of ability can devote their energies to the pursuit of science for science's sake; the environment is more favourable to development of the inventive faculty than of the peculiar talent for conducting abstruse researches in an exact science. Add to this the limited facilities for acquiring chemical knowledge in the New World, and the distance of amateurs from the European head-centres of learning, and it is certainly noteworthy that American chemists combined to form associations for mutual improvement and the advancement of their calling at so early a period.

A fourth attempt to establish a chemical society was made at New York City in 1876; the organisation was at first somewhat restricted in its plan, but in 1892 a change in its constitution was effected, which broadened its scope, and it now forms a strong, influential, and truly national society. Its 1106 members, working in nine chartered sections, represent forty-seven states and territories, besides several countries of Europe, South America, and distant Australia. Its Journal, comprising 1150 pages annually, is an authoritative medium for the preservation and diffusion of the researches made in the United States, and its annual meetings, held in diverse localities, strengthen the bonds which unite its members in good fellow-

ship, and in the pursuit of their common profession. *Long may the American Chemical Society continue in its prosperous career!*

PROCESS FOR SEPARATING AND DISTILLING BROMINE FROM A MIXTURE OF ALKALINE CHLORIDE AND BROMIDE.

By H. BAUBIGNY and P. RIVALS.

THE decomposition of bromides by the action of a solution of copper sulphate and potassium permanganate does not permit us to collect the bromine or to determine it directly by diffusing when operating in a vacuum. It partly attacks the grease which serves as a luting. We can therefore know the weight, having previously determined that of the chlorine and the sum of the weights of the two elements. But every process of indirect determination is imperfect, especially when the body in question is the smaller in quantity.

We have therefore sought to remove mechanically the bromine from the liquid, either by ebullition or by a current of air.

The method of ebullition in which the watery vapour formed has the object of expelling the free bromine has also revealed the inconveniences involved. The first is the formation of enormous volumes of condensed water. Secondly, the ebullition causes rapid variations of the liquid volumes. Now the alkaline chlorides, stable at 100°, if the solution is not too strongly charged with salts of copper, are decomposed if the concentration exceeds certain limits. The use of a current of air, on the contrary, has permitted the easy solution of the question, and can be used at any temperature. — *Comptes Rendus*, No. 15, October 11, 1897.

NOTICES OF BOOKS.

Annali del Laboratorio Chimico Centrale delle Gabelle, pubblicati da Vittorio Villavecchia. Vol. III. Roma, 1897. 242 pp. 8vo.

THE first volume of the *Annali* of the Central Chemical Laboratory of the Customs was issued in 1891, and covered the period from 1886-89; the second volume bore the date 1893; the present volume chronicles the work done in the years 1890-96. More than four thousand eight hundred analyses were made in the year 1896, the total number for the period 1890-96 being 24,913. The substances examined included wines, vermouth, beer, spirits; fixed, mineral, and volatile oils; mineral waters; coffee, chocolate, spices, confectioners' preserves, candy, &c.; condensed milk; effervescent magnesium citrate, salts of Stasfurt; vegetable and mineral colouring substances, varnishes, inks, minerals, cements, cereals; fats, soaps, waxes; and a variety of chemical and pharmaceutical products.

The volume contains records of the following researches:—

I. "On the Substance found in Sesame Oil, and on the Relation which it bears to the characteristic Colour Reaction of this Oil." By V. Villavecchia and G. Fabris.

II. "On the Composition of Italian Flour." By G. Fabris and O. Severini.

III. "On the Paper Mulberry." By M. Tortelli.

IV. "Soap Analysis." By R. Moreschini.

V. "On Illuminating Oil and its Consummation in Italy." By A. Volpi and R. Ruggieri.

VI. "On Dulcina, or Paraphenetol Carbamide, and a Method of Recognising it." By R. Ruggieri.

VII. "Analysis of Candies." By A. Bianchi and O. Severini.

VIII. "On the Chemical Composition of some Greek Wines." By R. Moreschini.

IX. "Researches on *Digras* (Fish Oil)." By M. Tortelli.

X. "On the Estimation of Glycerin in Sweet Wines." By G. Fabris.

The volume is well printed, but the absence of running-headlines makes it difficult to find the several essays.

The monograph on the paper-mulberry is accompanied by a plate.

H. C. B.

CORRESPONDENCE.

ARGON AND HELIUM.

To the Editor of the Chemical News.

SIR,—I would like to call the attention of chemists who are working at the gases argon and helium to the fact that there is near Mallow, Co. Cork, a mineral spring which evolves large quantities of nitrogen. According to an analysis by Prof. Daubeny the constitution of the evolved gas is—

Nitrogen	93.5 parts
Oxygen	6.5 "

100.0

Near Hillebrook, in the parish of Killrerin (Barony of Clare), is another spring, of which the following analysis of the evolved gas was made:—

Analysis of a Wine Pint of Water in May, 1842.

Nitrogen	0.59 cub. ins.
Carbon dioxide .. .	1.0 "

Most probably the above-mentioned gases contain argon or even helium.—I am, &c.,

C. P. FINN.

The Yorkshire College, Leeds,
November 1, 1897.

THORIUM ACETYL-ACETONATE.

To the Editor of the Chemical News.

SIR,—If my request is not unreasonable I should feel extremely favoured if anyone could inform me whether the acetylacetone referred to in the article on "Thorium," which appeared in No. 1971 of the CHEMICAL NEWS (vol. lxxvi., p. 110), is the ordinary acetone or no.

If it is not acetone I am at a loss to understand to what the author refers, and am unable to find any information on the subject either in "Dict. Chem." Thorpe and Muir, or Gmelin or Watts.

Presuming acetone to be meant by acetylacetone I have carefully followed the process, but failed to prepare the acetylacetone of thorium mentioned therein.

Taking an exceptionally pure thorium salt, which was prepared for a special purpose by repeated precipitations, as "hypo," "oxalate," and ultimately re-crystallising the double ammonium salt several times, I converted it into the chloride, precipitated with ammonia, washed, and removed excess of water by pressure.

This purified hydrate was suspended in dilute alcohol (1 ab. to 5 aq.), mixed with acetone, and evaporated to dryness on water-bath. The process failed, however, to yield any substance soluble in chloroform, nor were repeated trials more successful.

As the commencement of paragraph 2 is slightly ambiguous I tried a further experiment by acting on the $\text{Th}(\text{HO})_4$ with peroxide of hydrogen before the acetone treatment, after which I carefully followed the process as given by M. Urbain. Obtaining no better results I am

forced to conclude that acetylacetone is some unusual substance beyond the knowledge of an ordinary inorganic chemist, and have therefore ventured to trouble you in this matter.—I am, &c.,

ALLANITE.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 16, October 18, 1897.

Impurities of Crude Copper.—M. Schlagdenhauffen.—The treatment by nitric acid of the sublimate in one of the author's tubes yields on analysis all the characters of selenious acid.

Electrolytic Conductivity of Trichloroacetic Acid.—Paul Rivals.—Not adapted for useful abstraction.

The Mean Molecular Weight of the Soluble Matter in Seeds during Germination.—M. Maquenne.

No. 17, October 26,

Novel Procedure for obtaining Instantaneous Radiographs.—G. Séigny.—Following the indications of Dr. Max Lévy the author takes a very thin plate of glass, which he coats on both sides with silver gelatino-bromide and then allows it to dry in this emulsion. He prepares meantime two flexible screens of cloth with violet calcium suspended in celluloid. When the screens are dry they are applied to each side of the double emulsion plate, the whole being then placed in a frame exerting a pressure on the surfaces by means of two plates of cardboard, and proceed as usual.

A New Bianodic Vessel for Red Phosphorescence. G. Séigny and E. Gundelag.—The authors have prepared the glass for these vessels by incorporating with coloured glass transparent and not fluorescent powdered albumen and calcium carbonate, or preferably didymium chloride. The glass thus prepared has the following properties:—1. Its fluorescence is in red and not green. 2. It emits twice as many X rays as the ordinary glass. 3. The fluorescence which it excites upon the screen is more brilliant and of yellowish green colour.

Researches on Saline Solutions. Lithium Chloride.—Georges Lemoine.

On Basic Magnesium Salts.—M. Tassilly.—The researches of various *savants* on magnesium oxychlorides and the author's own on the oxybromide tend to establish an approximation between this metal and zinc as regards the basic salts.

Separation and Direct Determination of the Chlorine and Bromine contained in a Mixture of Alkaline Salts.—H. Baubigny and P. Rivals.

Some Compounds of Metallic Acetates with Phenylhydrazine.—J. Moitessier.—Phenylhydrazine forms with metallic acetate of the magnesium series compounds analogous to the phenylhydrazinic chlorides, bromides, iodides, and nitrates. The author has prepared the zinc, cadmium, manganese, cobalt, and nickel phenylhydrazines by heating in the water-bath a mixture of phenylhydrazine in an alcoholic solution and of a pulverised metallic acetate.

Methods of Determining Diabetic Sugar.—F. Landolph.—1. The polaristrobometer alone indicates the real quantity of active diabetic sugar. 2. The coefficient of reduction gives double and even threefold the polaristrobometric sugar. 3. Fermentation shows a very vari-

able quantity of sugar according to the duration of the fermentation.

Optical and Reductive Power of the Flesh of Flies.—F. Landolph.—The author finds that a filtered solution of the flesh of flies obtained by trituration with cold water is opalescent and strongly levo-rotatory. Its reductive power is certainly greater than that of diabetic sugar. The reductive power of the aqueous extract of spiders is also very high.

Journal de Pharmacie et de Chimie,
Series 6, vol vi., No. 7.

On the Question of Matches. Phosphorism.—A. Riche.—A long paper, to be continued, not suitable for abstraction.

Detection of Acetone in Urine.—A. Mallat.—In 1896 the author published the method of detecting acetone in urine, which consists of precipitating 100 c.c. of urine with 10 c.c. of subacetate of lead, then distilling a portion of this liquid with caustic soda, then adding to a small portion a solution of iodine and iodide of potassium. On reversing the test-tube once, the presence of iodoform is shown by the smell and the cloudy appearance of the solution, indicating that acetone was present in the urine. M. Bretet, who has used this method, has modified it in the following manner:—He does not treat the urine with subacetate of lead, but he distills it in the presence of tartaric acid; he stops the operation when about one-tenth has been distilled over. Then, in two test-tubes, he makes the mixture of caustic soda and of iodine in iodide of potassium, and thus obtains an absolutely clear and limpid reagent. To one of these tubes he adds the distilled urine under examination, and the second tube he uses as a check. The slightest formation of iodoform causes a cloudiness in the tube, and denotes the presence of the smallest traces of acetone.

Composition of Potatoes.—M. Balland.—The author finds that 3 kilograms of potatoes before or after cooking, equal to about 1200 grms. of fried potatoes or 700 grms. of desiccated potatoes, contain the same amount of nitrogenous and amylaceous matters as 1 kilogram of ordinary white bread.

Assay of Alloys of Copper and Nickel.—A. Riche.—The use of coins made of an alloy of copper and nickel in two of the French colonies, has rendered necessary at the Mint a rapid and exact method for the analysis of these alloys. They are not of the same composition: one containing 25 per cent of nickel, and the other only 15 per cent. The alloys being binary, it is only really necessary to determine one of the metals—the copper, for instance. But as the nickel is the most expensive of the two metals, it is preferable to estimate it directly rather than to deduce its weight by difference. The operation is carried out as follows:—About 1 grm. is attacked with the smallest quantity possible of nitric acid on a sand-bath; when finished, add a little water containing 5 or 6 drops of sulphuric acid and evaporate to dryness. Take up with water still containing a little sulphuric acid, and again evaporate to dryness to make sure that all the nitric acid is driven off; re-dissolve in water with a little sulphuric acid, and pour into an electrolytic crucible, filling it about one-third full. The copper alone is deposited. The remaining solution is saturated with ammonia and electrolysed with three Daniell cells. This brings down the nickel, and the whole process gives very concordant results. The difference between the total weights found and 100 represents the impurities—consisting of sesquioxide of iron, also oxides of manganese and aluminium—in the nickel. In the case of a bronze coin, 475 m.grms. of copper were deposited in four hours with a current of half an ampere.

No. 8.

On Antimonic Acids and Antimonates.—M. Delacroix.—Frémy described two antimonic acids and

their salts, but many chemists will not admit the existence of more than one. M. Delacroix has taken up the study and is convinced that there are really two: pyroantimonic acid, giving acid pyroantimonates and neutral salts; and orthoantimonic acid, giving acid, neutral, and basic salts. The solution of pyroantimonic acid was prepared from the hydrate by pouring SbCl_3 into 20 or 25 times its weight of cold water, or by treating SbCl_3 (previously dissolved in a little water), in the same manner, then saturating with chlorine. The excess of chlorine is driven off by a current of air directed on to the mixture. After standing an hour or two, the hydrate is transferred to a filter, washed a little, and pressed strongly between blotting-paper. It retains a little HCl very tenaciously, but not more than if it had been washed for a much longer time. The hydrate is then placed in cold water, in which it slowly dissolves. The solution, which is colourless, is evaporated to dryness and calcined, forming Sb_2O_4 , from the weight of which the amount of anhydride is calculated. Pyroantimonic acid, when warmed for some time at 100° , becomes transformed into the more basic orthoantimonic acid. The same transformation takes place spontaneously at the ordinary temperature in a few days. The solutions thus prepared are slightly opalescent. The author intends studying the basic orthoantimonates and the neutral pyroantimonates, which are new salts.

MISCELLANEOUS.

The Commercial Development Corporation, Limited.—We are officially informed that letters of allotment and regret of the above Company have been posted.

Society of Arts.—The Society of Arts will commence its Session (the 144th from its foundation in 1754) on the 17th inst. with an address on "The Colonies: their Arts, Manufactures, and Commerce," by Major-General Sir Owen Tudor Burne, G.C.I.E., K.C.S.I., the Chairman of the Society's Council. There will be four meetings on successive Wednesdays before Christmas, at which papers will be read by Prof. James Douglas, on "The Progress of Metallurgy and Metal Mining in America during the last Half Century"; by Prof. Leonard Waldo, D.Sc., on "The American Bicycle—the Theory and Practice of its Making"; by Bennett H. Brough, on "The Stockholm Exhibition of 1897"; by Samuel Rideal, D.Sc., on "The Purification of Sewage by Bacteria." The first course of Cantor lectures will consist of three lectures, to be given on Monday evenings, commencing on the 29th inst., by Dr. Eugene F. A. Obach, F.C.S., on "Gutta Percha."

Imperial Institute.—A commercial reading-room, open free to the general public, has been established at the Institute, in the hope that it may be specially useful to mercantile men, manufacturers, &c., either resident in, or visiting the Metropolis. A considerable number of commercial and technical publications (British, Indian, and Colonial and Foreign) can be read or consulted in the Reading-room. In addition, the room will contain a trade circulars, information in regard to shipping, transit by rail, &c. Maps of different portions of the Colonies are likewise exhibited, and many maps or charts included in the collection provided in the Map-room of the Institute may be consulted on application. Works connected with the Colonies and India (such as Diplomatic and Foreign Consular Reports, Directories, Year-books and Handbooks, Government Gazettes, Market Reports, Prices Current and Statistics, Parliamentary Papers and Blue-books, Statistical Registers, Weather Reports, &c.) are available to the public visiting the Reading-room, on filling up application forms.—*Journal of the Society of Arts.*

NOTES AND QUERIES.

* * Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Soluble Essences.—Monazite.—1. Would anyone oblige me by informing me of the best manual on the distillation and manufacture of soluble essences, such as are used in the manufacture of aerated waters. 2. The appearance, &c., of monazite, and where specimens may be procured and at what cost.—C. F. F.

MEETINGS FOR THE WEEK.

WEDNESDAY, 17th.—Sanitary Institute, 8. (See p. 231).
Society of Arts, 8. Opening Address of Session by Major-General Sir Owen Tudor Burne, on "The Colonies—Their Arts, Manufactures, and Commerce."

THURSDAY, 18th.—Chemical, 8. "Decomposition of Camphoric Acid by Fusion with Potash or Soda," by A. W. Crossley, M.Sc., Ph.D., and W. H. Perkin, jun., F.R.S. "Experiments on the Synthesis of Camphoric Acid," by W. H. Bentley, B.Sc., and W. H. Perkin, jun., F.R.S. "Action of Magnesium on Cupric Sulphate Solution," by Frank Clowes, D.Sc., and R. M. Caven, B.Sc. "Properties and Relationships of Dihydroxy-Tartaric Acid," by H. J. Horstman Fenton, M.A.

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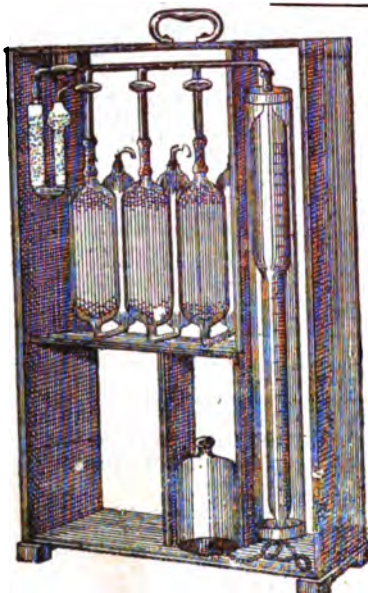
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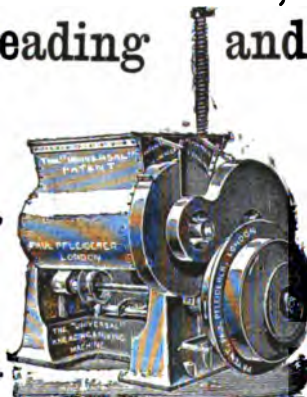
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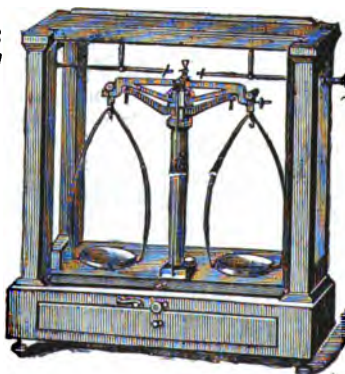


FIG. 3a.

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THE CHEMICAL NEWS.

Vol. LXXVI., No. 1982.

ON THE ESTIMATION OF COPPER AS IODIDE.

By M. WILLENZ.

THE volumetric estimation of copper as iodide, though old and well known, is being gradually abandoned, not having given, according to a great number of writers, such satisfactory results as were expected. For the last few years, however, chemists have again taken up the study of this question, and the estimation of copper as iodide is being revived, and is even officially adopted in some countries, notably the United States of America.

This method is, in fact, simple and rapid, and only requires the use of ordinary reagents. We were desirous of testing the accuracy and value of the process in question, and we now submit our results to the readers of the *Review*.

We have attempted to apply this method to cupreous pyrites, minerals containing at the most 6 per cent of copper, and we are able to state that the results obtained by following the plan we are about to describe have been very favourable, provided always that certain precautions are taken, and that the conditions of working are always identical.

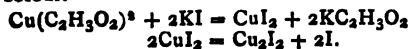
We proceed as follows:—

A solution of hyposulphite of soda is prepared, containing 9.92 grms. of the crystallised salt ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{Aq}$) per litre; this makes a $\text{N}/25$ solution, of which each c.c. is equivalent to 0.00252 grm. of copper. But as the hyposulphite is hardly ever pure, it is absolutely indispensable to control the titration by means of metallic copper, and further, as the hyposulphite alters more or less rapidly, and as its titration value is always going back, it becomes necessary to verify it before each series of experiments, and for this purpose it is well to keep a standard cupric solution.

To prepare this solution we dissolve 10 grms. of electrotype copper in a mixture of equal volumes of water and nitric acid, $\text{D} = 1.4$. When all the copper is dissolved, we boil for a long time until all the nitrous vapours are driven off, let cool, and make up to 1 litre.

To determine the titration value of the hyposulphite, we take 10 c.c. = 0.1 grm. of the cupric solution, in an Erlenmeyer flask of 200 c.c. capacity, having a mark at 50 c.c.; add dilute ammonia drop by drop until the blue precipitate of the basic salt no longer forms, then add dilute acetic acid drop by drop to re-dissolve the basic salt, and finally make the solution strongly acid with 5 or 6 c.c. of concentrated acetic acid; dilute to 50 c.c., and add 10 c.c. of a 10 per cent solution of potassic iodide, free from iodate, = 1 grm. KI.

The addition of potassic iodide causes the formation of cupric iodide, which immediately splits up into cupreous iodide and free iodine; the latter gives the solution a brown colour.



The result of this is that 63 parts of copper require 332 parts of iodide of potassium, or, in other terms, 0.1 grm. of copper requires 0.527 grm. of iodide; but as the reaction occurs but slowly when we take only the theoretical quantity, it is better to add 0.1 grm. of iodide, especially as an excess, even when considerable, has no interfering influence on the final results. We allow the iodide to react for two minutes, but no longer, and titrate with the hyposulphite, which is run in until the liquid

takes a clear yellowish brown tint; we then add a little starch solution, and while agitating we add more hyposulphite. As soon as the colour of the iodide of starch begins to change to a dirty greyish violet, we add the hyposulphite by only a drop at a time, shaking thoroughly after each addition.

We have remarked, in fact, that the violet tint will still persist even after all the iodine has been transformed into sodic iodide, but violent agitation will make it disappear, and the straw colour, due to cuprous iodide, shows itself, thus indicating the end of the titration. To get concordant results it is necessary always to stop at the same point; this is not difficult after a little practice.

When carried out in this manner the method gives perfectly concordant and very satisfactory results. We have never noticed the re-appearance of the blue colour, even after standing several hours.

We give below several titrations made with pure cupric sulphate, $\text{CuSO}_4 \cdot 5\text{aq.}$, containing 25.421 per cent of metallic copper. The aqueous solution of the salt was acidulated with 0.5 c.c. of nitric acid, $\text{D} = 1.4$, then treated with ammonia and acetic acid as above, diluted to 50 c.c.; 10 c.c. of 10 per cent iodide of potassium was then added, the whole let stand for two minutes, and then titrated,—

1 c.c. $\text{Na}_2\text{S}_2\text{O}_3 = 0.00249376$ grm. Cu.

Application of the Method to the Estimation of Copper in Pyrites.—Ten grms. of finely powdered and dried pyrites are placed in a tall narrow beaker, 8 c.c. of water and 2 c.c. of concentrated sulphuric acid are added. Cover the beaker with a watch-glass, and add nitric acid, $\text{D} = 1.4$, in small quantities, until there is no longer any effervescence; this will require about 25 to 30 c.c. of HNO_3 , then add 3 c.c. more of H_2SO_4 . Boil well, over a naked flame for preference, and after a few minutes take off the watch-glass, and, without washing it, place it temporarily on one side. Continue boiling, and at the same time keep the beaker constantly turning—this is an indispensable precaution—until the mass, becoming thicker and thicker, will no longer run, but becomes quite pasty. Treat it now with hot water and replace the watch-glass, boil for a short time and allow to cool: make up to half a litre and filter.

The residue, which is quite free from copper if the operation has been properly conducted, consists of silica, sulphur, PbSO_4 , (BaSO_4 , CaSO_4). This operation requires a certain amount of skill, and special care must be taken lest the sulphur set at liberty envelops the unattacked particles of mineral, and thus prevents them being acted upon, but with a little practice this is easily avoided; the operation requires but half an hour at the most.

Take 100 c.c. = 2 grms. of pyrites, of the sulphuric solution thus obtained, in a conical flask, and add a few c.c. more of H_2SO_4 ; boil, and without stopping the heat add, little by little, a warm concentrated solution of hyposulphite of soda.

The addition of the first few drops produces no change, but there is soon a lively disengagement of sulphurous acid gas, and the liquid becomes cloudy, then successively greenish, brownish, chocolate-brown, and finally black, without any precipitate appearing. But by keeping up the boiling the precipitate comes down, agglomerates, and falls to the bottom of the flask, while the supernatant liquid becomes limpid.

This precipitate consists of sulphides of copper, arsenic, antimony, and tin, mixed with free sulphur, but it must be remembered that the last three sulphides are only partially precipitated when not in the presence of a great excess of acid; it may even happen that they are not precipitated at all if the bases are present in small quantities only. The other bases of the copper group are not precipitated,* the iron is reduced to the state of ferrous

* It is, however, worthy of remark that bismuth is precipitated under these conditions, but we have never come across this metal in pyrites.

Grms. CuSO ₄ aq.	Cu calculated.	C.c. of Na ₂ S ₂ O ₃ used.			Cu found.	Difference.
		I.	II.	Mean.		
0.10	0.025421	10.15	10.20	10.18	0.025386	-0.000035
0.15	0.038131	15.35	15.30	15.33	0.038229	+0.000098
0.20	0.050842	20.40	20.40	20.40	0.050873	+0.000031
0.25	0.063552	25.50	25.50	25.50	0.063591	+0.000038
0.30	0.076263	30.55	30.60	30.58	0.076259	-0.000004
0.35	0.088973	35.65	35.65	35.65	0.088902	-0.000071
0.40	0.101684	40.75	40.70	40.73	0.101571	-0.000113
0.45	0.114394	45.85	45.90	45.87	0.114389	-0.000005
0.50	0.127105	51.05	51.00	51.02	0.127232	+0.000127
0.60	0.152526	61.20	61.20	61.20	0.152618	+0.000092
0.70	0.177947	71.45	71.40	71.42	0.178104	+0.000157
0.80	0.203368	81.50	81.50	81.50	0.203241	-0.000127
0.90	0.228789	91.90	92.00	91.95	0.229301	+0.000512
1.00	0.254210	102.20	102.20	102.20	0.254862	+0.000652

salt; the aluminium, zinc, manganese, nickel, and cobalt remain in solution.

The precipitate is washed several times with boiling water, first by decantation, and afterwards on the filter. When obtained under the above-mentioned conditions it oxidises very slowly, and may be washed with pure water without any risk, but the washing should be done rapidly. When the wash waters no longer precipitate BaCl₂, we take the filter and its contents out of the funnel, squeeze it gently between blotting-paper to get rid of most of the moisture, then place it, point uppermost, in a porcelain crucible. Heat very gradually, and when all the water is driven off increase the temperature, and finally roast, so that all the sulphur is burnt, and the arsenic, antimony, and tin volatilised; there then remains in the crucible nothing but a mixture of oxide and sulphide of copper.

This is dissolved in 1 c.c. of a mixture of equal volumes of water and nitric acid, D=1.4, and boiled to completely drive off all nitrous fumes; transfer to a 200 c.c. Erlenmeyer flask, with a mark at 50 c.c.; add dilute ammonia, drop by drop, until the precipitate of the basic salt is no longer formed; this is re-dissolved in weak acetic acid, acidulated strongly with 5 or 6 c.c. of strong acetic acid; 10 c.c. of iodide of potassium are then added, and the whole left for two minutes; it is then titrated with hyposulphite, observing the precautions mentioned above.

As we have already said, this method gives very satisfactory results; it is easy to perform, and requires but very little time. Examples:—

1 c.c. Na₂S₂O₃ = 0.0025 grm. Cu.

Grm. Cu.	Grm. As.	C.c. of Na ₂ S ₂ O ₃ .	Cu found.	Difference.
0.0972	+0.4013	39.0	0.0975	+0.0003
0.0647	+0.4735	26.0	0.0650	+0.0003
0.0892	+0.7102	35.75	0.08937	+0.00017
0.1241	+0.8284	49.8	0.1245	+0.0004
0.2004	+1.4672	80.25	0.20062	+0.00022

Pyrites.	Contents of As, per cent.	Cu found	
		By Fresenius's method, per cent.	By this method, per cent.
I.	0.320	3.267	3.272
II.	0.024	0.069	0.081*
III.	0.062	4.584	4.601
IV.	0.182	0.074	0.081*
V.	traces	1.823	1.872
VI.	0.307	5.749	5.771
VII.	Not determined	2.218	2.330

* Five grms. of material used.

It is evident that this method can be applied equally to copper ores properly speaking, to bronzes, alloys, &c., but in these cases the quantity taken for assay must be less, and the solution of hyposulphite used may be more concentrated, a decinormal solution for instance; however

we prefer a weak solution, the inherent errors being then reduced to a minimum.—*Revue de Chem. Analytique*, Vol. v., No. 18.

ON THE DISSOCIATION SPECTRA OF MELTED SALTS.

ALKALINE METALS: SODIUM, LITHIUM, POTASSIUM.

By A. DE GRAMONT.

THE easiest dissociation spectra to study with the condenser spark are those of the salts of the alkaline metals, on account of their fusibility, stability, and simplicity.

To do this, it suffices to deduct from the total spectrum the metallic lines, to get the complete spectrum of the metalloid without having recourse to Plücker or Salet tubes. I therefore preferred commencing with the spectra of the common alkaline metals as they appear in the dissociation of their fused salts. They differ to a certain extent from those yet obtained, either with the pure metal and not much condensation, or with the fused salt and the coil only. Many of the lines, especially those in the most refrangible part of the spectrum, become wider and more diffused; the component parts of double lines being difficult to separate, no matter what dispersion is used, and appearing as fairly wide bands.

Most precise measurements of the wave-lengths of sodium will be found in the paper by MM. Eder and Valenta (*Deutschr. Kais. Akad. d. Wissenschaft*, vol. lxi., Vienna, 1894), who worked with the metal itself in an atmosphere of hydrogen and with a small condenser.

The spectrum of lithium has been measured with great accuracy by MM. Kayser and Runge (*Abhandlung Berliner Akad.*, 1890) in the arc, where the lines are the same as those now given and described.

I have established the lines peculiar to each metal under the following conditions:—1. By subtracting from the complete spectrum of a salt the lines of the combined metalloid, and repeating the experiment with several salts of the same metal. 2. By observing the spectrum of a carbonate, where the carbon lines appear only under special conditions. This has been recently proved (*Comptes Rendus*, vol. cxv., July 19 and 26, 1897). The dissociation of the salt is more or less easy to effect according to its nature; it is easy with chlorides, bromides, iodides, sulphides, sulphates, phosphates, &c.; it is more difficult with carbonates and fluorides, which require a much higher temperature and a greater difference of potential. The measurements of the wave-lengths here given were obtained with a direct-vision spectroscopic with two compound prisms, easily doubling the D line of sodium (Na α), which appeared to be one division of the micrometer apart; these divisions, with a little practice,

could be read to one-tenth. These values were compared with the normal solar spectrum photographed by Rowland. I have kept the alphabetical description for Na and K given by M. Lecoq de Boisbaudran in Plate V. of his "Atlas of Luminous Spectra."

Sodium.

δ	616.1	Strong, bright.
	615.5	" "
α	589.6	Intense. "
	589.0	" "
β	568.8	Strong, bright.
	568.3	" "
	567.5	Faint. "
	567.0	" "
ϵ	515.5	Fairly strong.
	515.1	" "
γ	498.3	Easily seen, very diffuse, almost joined.
	497.9	" "
	467.2	Faint, diffused.
	467.0	Fairly bright, diffused.
	454.5	Faint, diffused.
	449.9	Fairly bright, diffused.

Practically, and above all in the presence of metalloids, the spectrum of sodium is reduced to three double lines, bright and characteristic, Na δ , Na α (D of Fraunhofer), and Na β , each one being seen as a single line in a monoprismatic instrument with a medium slit. Na ϵ comes next in strength. The value I give it appears rather high (MM. Kayser and Runge give 515.37 and 514.92). The remaining double lines are much weaker and indistinct, almost forming a single diffuse band. Those marked * could not be split up on account of the condenser used.

Lithium.

	670.8	Strong, bright.
	610.3	Very strong.
	497.2	Strong.
	460.3	Strong, wide, diffuse.
	427.3	Well marked.
	413.2	Well marked, very diffuse.

A spectrum of such simple character, of six lines only, and so easily seen, renders the use of lithium salts particularly favourable for the study of the spectra of metalloids. The first four lines are always seen with the disruption discharge, either on the metal or on the solution of one of its salts, but the relative intensities of the lines vary in the two cases. 427.3 has never been seen before except in the electric arc; it is here very distinct, even with a small condenser. The existence of 413.2 was foretold by M. Lecoq de Boisbaudran, who calculated its existence from theoretical considerations, before discovering it in a concentrated solution; it is much easier to detect in melted Li_2CO_3 . In the condenser spark it is, on the other hand, very wide and cloudy.

The principal papers relating to the spectrum of potassium are based on experimental conditions very different to those under which I have worked; that is to say, the action of a strong condensation effecting the complete dissociation of a melted salt. By this means I obtained, united in one spectrum, the different lines which have been detected singly, by very different methods, and between which we can notice the relations between relative intensities; these do not resemble the corresponding lines found in the preceding researches.

M. Lecoq de Boisbaudran worked with a non-condensed spark on fused sulphate of potassium; Sir William Huggins and MM. Eder and Valenta with slight condensation at the ordinary temperature; Profs. Liveing and Dewar and MM. Kayser and Runge, in the electric arc, where the spectrum of potassium is notably less rich in lines than in the spark. The electrical and optical arrangements here used were the same as those in my previous experiments already described.

Potassium.

δ	769.9	Difficult to see.
	766.6	" "
γ	693.9	Fairly strong.
	691.4	Well marked.
	630.8*	" "
	624.55*	Easily seen.
	611.75†	Very well marked.
	583.2	Strong.
α	581.1	Fairly well seen.
	580.1	Strong.
	578.3	Very well marked.
	551.5*	Weak.
	536.0	Easily seen, diffuse.
	534.4	Fairly well seen, diffuse.
β	534.0	Easily seen, diffuse.
	532.35	Fairly well seen, diffuse.
η	511.3	Fairly well seen, diffuse, and almost confounded with one another.
	509.9	" "
	505.1‡	Weak.
	500.7	Weak.
	485.55§	Fairly well seen.
	485.1§	" "
ζ	482.9	Fairly strong.
	465.2	Fairly well seen.
	460.6¶	" "
	450.65¶	Weak.
	446.6	" "
	438.8¶	Fairly well seen.
	430.95	" "
	430.65¶	" "
	426.4§	Fairly strong.
	422.55	Fairly well seen.
	422.25	" "
	421.0	Weak.
ϵ	418.55¶	Fairly strong, wide.
	404.55	Strong, very wide, diffuse.

* Seen by Sir William Huggins only.

† Seen by Sir William Huggins, afterwards by M. Lecoq de Boisbaudran only.

‡ Seen by M. Lecoq de Boisbaudran only.

§ Seen by Profs. Liveing and Dewar only.

|| Seen by MM. Eder and Valenta only.

¶ Seen by M. Lecoq de Boisbaudran, afterwards by MM. Eder and Valenta only.

Several lines in the violet, detected by MM. Eder and Valenta by means of photography, which is more sensitive than the eye in this part of the spectrum, were not visible to me.

To get greater accuracy with regard to wave-lengths, we can refer to the memoir by MM. Kayser and Runge on the arc spectrum of potassium (*Abhand. Berliner d. Akad.*, 1890), and for the lines which do not show in the arc, to the work of MM. Eder and Valenta already quoted. All these observers had at their disposition either a Rowland's apparatus or a spectrograph—instruments which are not to be found at the Faculté des Sciences in Paris. The approximation here given, however, amply suffices for the identification of the lines in any current research in a chemical laboratory.

The classic red lines, K δ , situated at the end of the spectrum, always rather diffused, are only visible when working with instruments of little absorption; further, they are not mentioned in the work of Sir William Huggins or of M. Thalén. The double line, K γ , on the contrary, is very bright and characteristic; the second, or more narrow line, is slightly fainter than its companion. There are, again, three lines in the red—630.8, 624.55, and 611.75—which, though mentioned, but considered to be doubtful by Sir William Huggins, were either not seen by subsequent observers, or else attributed by them to impurities. However, the strongest and most refrangible, 611.75, was seen, though very faintly, by M. Lecoq de Boisbaudran, using a simple spark only, with pure fused sulphate of potash. MM. Eder and Valenta con-

sidered these rays as not belonging to potassium, but made no endeavour to define their origin. I have always found them in company with the double line $K\gamma$ in melted salts of potassium, when using a sufficient condensation. I repeat, that I have always succeeded in obtaining them eventually with complete absence of lines of other foreign matters; I consider them, therefore, as characteristic of the spectrum of dissociation of potassium in its salts.

The brightest group is $K\alpha$, in the green, and appears to show the most sensitive reaction of potassium in the spark. $K\beta$ is fainter and diffuse, and the two middle lines cannot be separated with a single prism. $K\gamma$ is still fainter, and more diffuse than under ordinary conditions; it is easily seen as a single band about 510.5 .

In the blue, K is, on the contrary, sensibly stronger and becomes characteristic: this line is, however, entirely missing in the arc.

In the same manner, 426.4 in the indigo, and 418.55 in the violet, become more intense with condensation, the augmentation of which makes itself particularly felt in this region of the spectrum. $K\delta$, the last line visible, is a double line which, in the condenser spark, becomes transformed into a strong diffuse band which cannot be divided; it is very bright and characteristic of potassium. — *Bull. Soc. Chim.*, Series 3, vols. xvii.-xviii., Nos. 16-17.

THE COMBUSTION OF ORGANIC SUBSTANCES IN THE WET WAY.*

By I. K. PHELPS.

In a former paper (*Am. Journ. Sci.*, vol. ii., p. 70) I have shown that carbon dioxide may be estimated iodometrically with a fair degree of accuracy. Inasmuch as this method is not dependent upon the rate of flow or rapidity of generation of the carbon dioxide, it seemed possible that some advantage might follow its application to the determination of organic carbon, oxidised by liquid reagents.

Method of Oxidation by Potassium Permanganate.

The first experimental test in this direction was made with oxalic acid, which was oxidised according to the well-known reaction of potassium permanganate in the presence of sulphuric acid. The apparatus used was the same as that previously described in the iodometric process referred to above. It consisted, in the main, of an evolution flask and an absorption flask, properly connected. As an evolution flask, a wide-mouthed flask of about 75 c.m.^3 capacity was used. This was closed by a doubly perforated rubber stopper, carrying a separating funnel for the introduction of liquid into the flask and a glass tube of 0.7 c.m. internal diameter, which was expanded to a small bulb just above the stopper, to carry off the gas. This exit tube was joined by means of a rubber connector to a tube which passed through the rubber stopper of the absorption flask, which was an ordinary round-bottomed flask of 250 c.m.^3 capacity. This tube ended in a valve of the Kreider pattern (*Am. Journ. Sci.*, l., p. 132), which was enclosed in a larger tube, reaching nearly to the bottom of the absorption flask. The second hole of the stopper of this absorption flask was filled by a glass tube closed by a rubber connector and screw pinch-cock.

The barium hydroxide solution for use in the determination of the carbon dioxide was prepared by filtering a cold saturated solution of the commercial salt into a large bottle, which was connected with a self-feeding burette. The solution was standardised in the manner described in my former paper, by boiling with an excess of decinormal iodine solution in an ether wash-bottle. The short tube of the glass ground stopper of the bottle

was sealed to a Will and Varrentrapp absorption apparatus, which was charged during the operation with a solution of potassium iodide to prevent the loss of elementary iodine in the boiling; the long tube of the bottle was used as an inlet tube, and was closed externally by a rubber cap during the boiling. After cooling, the excess of iodine used was determined by titration with decinormal arsenious acid solution and the iodine lost calculated on barium hydroxide molecule for molecule.

Potassium permanganate was prepared for use by dissolving the commercial salt in water, and boiling this solution made acid with sulphuric acid, until free from carbon dioxide. Water was also prepared free from carbon dioxide by boiling distilled water until one-third had been driven off in steam and was kept until used in full-stoppered flasks.

For the first determinations of carbon, crystallised ammonium oxalate was weighed out and introduced into the boiling flask with 10 to 15 c.m.^3 of pure water and the flasks connected as described above with an appropriate amount of barium hydroxide solution (3 to 5 c.m.^3 in excess of the amount required to precipitate the carbon dioxide to be determined) in the absorption flask. The whole system was then evacuated with the water-pump to a pressure of 200 – 225 m.m. and the oxalate solution in the boiling-flask warmed. An excess of potassium permanganate solution was then run in through the funnel tube and the mixture warmed again, when the oxidation of the oxalate was shown by the carbon dioxide evolved. The carbon dioxide was completely set free by the introduction of 10 c.m.^3 of sulphuric acid ($1:4$) and was driven completely to the absorption flask by boiling for five minutes. During the passage of the gas into the absorption-flask, it was shaken frequently and was kept cool by standing in a dish of water and by pouring cold water over it from time to time. If, during the boiling, any fears are entertained as to the strength of the vacuum in the flasks, they may be easily allayed by opening momentarily the stopcock of the funnel-tube and noting the direction of the flow of water contained in the funnel. After the boiling was ended, the atmospheric pressure was restored by allowing air, purified from carbon dioxide by passage through potash bulbs, to enter through the funnel-tube of the boiling flask. Then the flasks were disconnected and the stopper of the absorption-flask with its attachments was removed, the valve and its tube being carefully washed free from barium hydroxide. A second stopper, which was provided with a separating funnel, and a Will and Varrentrapp absorption apparatus, containing water to serve as a trap, was inserted into the mouth of the absorption flask and the emulsion brought to the boiling point. Decinormal iodine solution was then run in through the funnel-tube in sufficient quantity to destroy the larger part of the excess of barium hydroxide, and the emulsion brought to the boiling-point again, after which iodine was again run in, but this time to the permanent red colour of the excess of free iodine. After cooling, this excess of iodine was determined by titration with decinormal arsenious acid solution. Thus, the excess of barium hydroxide used being determined by the iodine lost, the barium hydroxide used, now in the form of carbonate, was known, from which the carbon dioxide which precipitated this carbonate may be calculated.

The following results were obtained by this procedure.

TABLE I.

	Ammonium oxalate taken. Grm.	BaO ₂ H ₂ found. Grm.	BaO ₂ H ₂ found. Grm.	CO ₂ found. Grm.	CO ₂ calculated. Grm.	Error on CO ₂ . Grm.
1.	0.2522	0.7267	0.1170	0.1565	0.1561	0.0004+
2.	0.2542	0.7267	0.1113	0.1579	0.1574	0.0005+
3.	0.5020	1.4535	0.2417	0.3110	0.3108	0.0002+
4.	0.5058	1.3954	0.1753	0.3131	0.3131	0.0000±
5.	1.0033	2.6163	0.1955	0.6213	0.6211	0.0002+
6.	1.0003	2.5951	0.1836	0.6189	0.6192	0.0003–
7.	1.0010	2.6163	0.2037	0.6192	0.6197	0.0005–

* Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, Series 4, Vol. iv., No. 23, November, 1897.

In Experiments 5 and 6 a few drops of ammonia were added to the oxalate solution before running in the permanganate; in 3 and 7, the permanganate was treated to alkalinity with barium hydroxide; in the remaining experiments, 1, 2, and 4, the permanganate was slightly acid with the sulphuric acid used in its purification from carbon dioxide, as already described. The results obtained are good, and it is plain that the oxidation proceeded regularly, whether the first action of the permanganate was in the alkaline or slightly acid solution.

Jones (*Am. Chem. Journ.*, xvii., 539) has shown that formates may be determined volumetrically by titration with potassium permanganate in alkaline solution. In an attempt to determine formates by the process outlined above, the pure barium salt was used. This was prepared by treating the aqueous solution of formic acid with pure barium carbonate to neutrality and crystallising the product. It was proven pure by ignition and weighing in the form of carbonate.

In making determinations of carbon in this formate, weighed portions were introduced into the boiling flask, together with sodium hydroxide solution, which was taken in such quantity as to more than neutralise the acid in the potassium permanganate. Naturally, the sodium hydroxide must be free from carbonate—which was effected by treatment with an excess of barium hydroxide and filtering. An excess of potassium permanganate is then run into the flask and the solution heated to boiling. An excess of dilute sulphuric acid is introduced into the mixture, and the carbon dioxide thus set free completely driven over to the absorption flask and determined as before. Table II. shows results obtained by the process.

TABLE II.

Barium formate taken. Grm.	BaO ₃ H ₂ taken. Grms.	BaO ₃ H ₂ found. Grm.	CO ₂ found. Grm.	CO ₂ calculated. Grm.	Error on CO ₂ . Grm.
1. 0.5001	0.9302	0.1745	0.1939	0.1935	0.0004+
2. 0.5033	0.9.12	0.1402	0.1953	0.1947	0.0006+
3. 1.0002	1.6861	0.1793	0.3867	0.3870	0.0003-
4. 1.0059	1.6279	0.1093	0.3897	0.3892	0.0005+
5. 1.3750	2.2529	0.1820	0.5315	0.5320	0.0005-
6. 1.5028	2.4419	0.1754	0.5816	0.5814	0.0002+

These results show plainly that the carbon of formic acid may be determined accurately by the method outlined.

It was found incidentally that ammonia cannot take the place of the sodium hydroxide in this process, probably because the ammonia volatilises to the absorption flask during the boiling and is acted on by the iodine subsequently used, and is thus registered as barium hydroxide.

It is a well known fact that tartrates are oxidised by permanganates. I have found, however, that when tartaric acid is treated under the conditions of analysis outlined above in acid solution, the oxidation is incomplete; but that oxidation is complete if the tartrate is heated in a solution alkaline with sodium hydroxide and then acidified with sulphuric acid.

The tartrate used was a re-crystallised tartar emetic, dried at 100° C. The following results were obtained with such a tartrate by this process.

TABLE III.

Tartar emetic taken. Grm.	BaO ₃ H ₂ taken. Grms.	BaO ₃ H ₂ found. Grm.	CO ₂ found. Grm.	CO ₂ calculated. Grm.	Error on CO ₂ . Grm.
1. 0.5051	1.2450	0.1709	0.2756	0.2751	0.0005+
2. 0.5030	1.2226	0.1536	0.2743	0.2739	0.0004+
3. 0.7509	1.7355	0.1401	0.4094	0.4091	0.0003+
4. 0.7541	1.7430	0.1410	0.4111	0.4107	0.0004+
5. 1.0018	2.3456	0.2187	0.5458	0.5456	0.0002+
6. 1.0005	2.2435	0.1196	0.5451	0.5450	0.0001+

It seems possible to draw the general conclusion from the results recorded that organic substances which are

oxidised completely by the permanganate may be determined by the process outlined above. It will also be seen that the use of the rubber stopper in the boiling flask, with due care to prevent its contact with the solution, does not introduce an appreciable error.

Wanklyn and Cooper (*Phil. Mag.*, [5], vii., 138) and others have noted the fact that potassium permanganate, whether in acid or alkaline solution, will not oxidise all organic substances (acetates, for example), even at the boiling temperature. It is well known that a mixture of concentrated sulphuric and chromic acids has a much wider field of action in oxidising organic compounds than the permanganate. With hopes of applying this reagent more widely to the determination of organic carbon, the experiments about to be recorded were tried.

(To be continued).

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING OCTOBER 31ST, 1897.

By SIR WILLIAM CROOKES, F.R.S.,

and

PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolis Water Act, 1871.

London, November 10th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 182 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from October 1st to October 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 182 samples examined during the month all were found to be clear, bright, and well filtered.

The rainfall at Oxford during the month shows a great deficiency, only 1.22 inches of rain having fallen, while the average for the last 30 years is 2.75 inches, making a deficit of 1.53 inches. The total rainfall for the year now shows a deficit of 0.41 inch.

Our bacteriological examination of 253 samples taken by us have given the following results; we have also examined 72 other samples, from stand-pipes, special points, wells, &c., making a total of 325 samples in all:—

	Microbes per c.c.
Thames water, unfiltered (mean of 26 samples)	3547
Thames water, from the clear water wells of five Thames-derived supplies (mean of 124 samples)	51
Ditto ditto highest	434
Ditto ditto lowest	0
New River, unfiltered (mean of 26 samples)	397
New River, filtered (mean of 26 samples)	35
River Lea, unfiltered (mean of 26 samples)	1588
River Lea, from the clear water well of the East London Water Company (mean of 25 samples)	39

The filtration and settling appliances of all the Companies have been in a high state of efficiency during the past month.

The water of every Company has been specially tested for pathogenic organisms, but with negative results.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.
JAMES DEWAR.

ELECTROLYTIC SEPARATION OF NICKEL AND COBALT FROM IRON. APPLICATION TO THE DETERMINATION OF NICKEL IN STEEL.

By O. DURN.

THE accurate separation of nickel, and nickel and cobalt, from large quantities of iron presents notable difficulties. The great number of methods already published proves that no really satisfactory solution of the difficulty has been reached.

For some years nickel steels have taken an important industrial rank, whence an expeditious and precise method presents a certain interest. In his chemical study on the methods of analysis of irons and cast metals, Ad. Carnot gives the preference to the method of Rothe, and this method, which has been simultaneously devised in France by Hanrich, depends on the separation of ferric chloride in an acid solution by means of ether. Latterly Pinerua has modified this method. He uses, at a low temperature, ether saturated with hydrochloric acid.

It is easy to arrive at the same result by means of electrolysis. If we precipitate by ammonia in excess a ferric solution containing, *e.g.*, nickel, a part of this metal is carried down by the ferric hydrate. If we submit to electrolysis the ammoniacal liquid holding the precipitate in solution, we may obtain on the cathode the entire deposit of the nickel. The separation is not absolutely accurate. Almost always a minute quantity of iron is also deposited upon the cathode, but in suitable conditions this quantity ranges about 1 to 2 m.grms. when the iron present may reach 400 to 500 m.grms. For precise experiments it is necessary to make correction in the weight of the metal deposited, which is easily done by dissolving the deposit in hydrochloric acid and precipitation with ammonia after for oxidising.

The use of the nitric solution, which under the like conditions enabled M. Riche to separate copper from iron, presents certain inconveniences. It is the same with the hydrochloric solution. We obtain good results by operating on the sulphuric solution to which ammonium sulphate has been added. The following is the process:—The solution containing nickel and iron as per-salts, with the addition, if needful, of a slight excess of sulphuric acid, is evaporated to dryness, is then taken up in a minimum of water with the addition of 5 to 10 grms. ammonium sulphate, and heated until a clear solution is obtained. This liquid is poured whilst stirred into the crucible of Riche's apparatus, in which have been placed 60 to 70 c.c. of concentrated ammonia.

As a source of electricity we use two or three accumulators, arranged in tension so as to regulate the intensity of the current at the outset at from 1.5 to 2.5 ampères. Under these conditions the nickel is entirely deposited in less than four hours.

The method has been checked by means of standardised solutions of iron and nickel, some of the results being—

	Iron.	Ni (added).	Metal deposited.	Correction for iron.	Ni recovered.	Difference.
1.	404.3	29.8	30.8	1.0	29.8	0.0
2.	269.5	74.6	75.9	1.3	74.6	0.0
3.	269.5	149.2	150.1	0.8	149.3	+0.1

The same procedure is equally applicable to cobalt:—

4.	404.2	62.5	63.4	1.9	61.5	-1.0
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Determination of Nickel in Steels.—We attack from 250 to 300 m.grms. of the sample with aqua regia in a porcelain capsule. When the action is complete we add 1 c.c. of sulphuric acid, and evaporate until white fumes are produced.—*Comptes Rendus*, cxv., No. 11.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

THE following are the abstracts of papers received during the vacation, and published in the *Transactions*:—

96. "The Ethers of Camphoroxime." By M. O. FORSTER, Ph.D.

The *methyl ether* of camphoroxime boils at 181.5–182.5° under a pressure of 357 m.m.; it has the sp. gr. = 0.9631, and the specific rotatory power $[\alpha]_D = -13.05^\circ$ at 20°. It does not reduce an ammoniacal solution of silver nitrate, and dissolves in mineral acids without undergoing change. The *nitrate* crystallises from benzene in needles melting at 81–82°, and has $[\alpha]_D = -16.9^\circ$ in benzene; the *hydriodide* is amorphous, and melts at 157° with vigorous effervescence.

The *ethyl ether* (Nigeli) boils at 185° under a pressure of 336 m.m., and has the sp. gr. = 0.9470; the specific rotatory power $[\alpha]_D = -19.0^\circ$ at 23.5°.

The *benzyl ether* is a colourless oil, which on distillation is in part resolved into benzaldehyde and camphorimine, as represented by the equation—



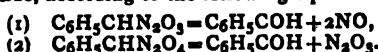
it has $[\alpha]_D = -16.4^\circ$ in alcohol, and forms an amorphous *hydriodide* which melts at 91°. Concentrated sulphuric acid resolves the ether into camphoroxime and the resinous hydrocarbon, $C_{14}H_{18}$, obtained on dissolving benzylic alcohol in concentrated sulphuric acid. Alcoholic hydrochloric acid eliminates α -benzylhydroxylamine from the ether; the *platinochloride* of this base forms golden-yellow scales, and does not melt below 250°.

The *acetyl derivative* of camphoroxime is a colourless liquid, and is completely converted into acetic acid and campholenitrile on distillation; it has the specific rotatory power $[\alpha]_D = -45.8^\circ$ in alcohol, and on treatment with cold phenylhydrazine yields symmetrical acetylphenylhydrazine and camphoroxime.

The *benzoyl derivative* crystallises from acetone in magnificent six-sided prisms, and melts at 88–90°; it has $[\alpha]_D = -40.7^\circ$ in alcohol, and yields benzanilide when heated with aniline. Cold phenylhydrazine gives rise to benzoylphenylhydrazine and camphoroxime. *Camphoroxime hydrobromide* melts and evolves hydrogen bromide at 174°; it has $[\alpha]_D = -35.8^\circ$ in alcohol, and is converted by glacial acetic acid into campholenitrile and hydrogen bromide. *Camphoroxime platinochloride* crystallises in transparent prisms which become opaque in the desiccator, and melts at 156.5° with vigorous effervescence; cold water regenerates the oxime. *Inactive camphoroxime* melts like the active modification at 118°; it crystallises from petroleum in diamond-shaped plates, and is racemic according to the classification recently suggested by Kipping and Pope (*Proc. Chem. Soc.*, 1897, p. 135).

97. "The Action of Nitrogen Trioxide and Tetroxide on Alcohols." Part I. By JULIUS BEREND COHEN, Ph.D., and HARRY THORNTON CALVERT, B.Sc.

The authors have found that when nitrogen trioxide or tetroxide dissolved in chloroform is allowed to act upon benzyl alcohol, that water is in both cases eliminated, and compounds of the formula $C_6H_5CHN_2O_3$ and $C_6H_5CHN_2O_4$ are probably formed, which rapidly decompose on standing into benzaldehyde, with the separation in the first case of nitric oxide, and in the second of nitrogen trioxide, according to the following equations:—



The latter substance, which may be termed benzylidene nitrosate, is decomposed by water into a compound of the formula $C_7H_7NO_3$, which is probably identical with a substance obtained by Lippmann and Hawliczek (*Ber.*, 1876, ix., 1463) by the action of nitric acid upon benzaldehyde. By the action of reducing agents it is converted into benzyl alcohol, benzylamine, and ammonia.

98. "The Action of Nitrogen Tetroxide on Ortho- and Para-nitrobenzylalcohol." By JULIUS B. COHEN, Ph.D., and WILLIAM H. HARRISON, B.Sc.

The authors have discovered a simple method for preparing the aldehydes corresponding to ortho- and para-nitrobenzylalcohol. The reaction consists in treating the alcohol with a small quantity of nitrogen tetroxide in presence of air. A nearly theoretical yield of these aldehydes, hitherto very difficult to prepare, has been effected by this method.

99. "The Action of Aromatic Amines upon Diacetyl-tartaric Anhydride." By JULIUS BEREND COHEN, Ph.D., and WILLIAM HUDSON HARRISON, B.Sc.

In attempting to prepare the isomeric toluidio-acetyl tartaric acids, by acting upon diacetyl tartaric anhydride with the isomeric toluidines, with a view to comparing their optical characters, the authors were unsuccessful; but obtained, on the other hand, by this reaction with different aromatic amines, a series of golden-yellow crystalline compounds. The formula of the aniline compound is probably $C_{16}H_{12}N_2O_3$, that of the paratoluidine compound $C_{18}H_{16}N_2O_3$, and of the α -naphthylamine compound $C_{27}H_{18}N_2O_3$. The constitution of these compounds has not yet been ascertained. The reaction in all cases is very complex, and the yield of the yellow substances very small.

100. "Studies on Citrazinic Acid." Part V. By W. J. SELL, M.A., and F. W. DOOTSON, B.A.

This investigation was commenced with the view of obtaining some evidence of the positions of the hydroxyl groups in citrazinic acid, by preparing the corresponding dichlorisonicotinic acid, and then replacing the chlorine atoms either by cyanogen or methyl, and thus by well-known methods obtaining one of the tricarboxy-acids whose constitution has been established. In the preparation of the dichlorisonicotinic acid by the interaction of phosphorus pentachloride on citrazinic acid, however, such a number of interesting substances were found to be produced that it was determined to publish this part of the work at once, leaving the remainder for a further communication. The following substances, amongst others, have been isolated, and are described in the paper:—

(1) Chlorhydroxyisonicotinic acid; (2) dichlorisonicotinic acid; (3) tetrachlorisonicotinic acid chloride; (4) $\alpha\beta\alpha'\beta'$ -tetrachlorisonicotinic acid; (5) tetrachlorpyridine; (6) pentachlorpyridine; (7) pentachlorpicoline.

101. "The Condensation of Chloral with Resorcinol." Part II. By J. T. HEWITT, M.A., D.Sc., and FRANK G. ROPE.

In an earlier paper (*Trans.*, 1896, lxix., 1265) the view was expressed that the substance of the formula $C_{14}H_{10}O_5$, obtained by the condensation of chloral

hydrate with resorcinol, was a lactone of 2:4:2':4'-tetrahydroxydiphenylacetic acid. The authors had overlooked a paper by Michael and Comey (*Amer. Chem. Journ.*, 1883-4, v., 350), in which the formula $C_8H_6O_3$ was attributed to the compound in question. The determination of the molecular weight by the lowering of freezing-point of a phenolic solution gave as result 232; the values required by the formulae $C_8H_6O_3$ and $C_{14}H_{10}O_5$ being 150 and 258 respectively. The analyses of the acetate and benzoate have further confirmed the authors' views that the compound possesses three hydroxyl groups. In addition to this, it has been found that a red salt is precipitated when an excess of sodium ethylate solution is added to an absolute alcoholic solution of the lactone: the salt was found to contain 22.10 per cent of sodium, whilst the formula $C_{14}H_7O_5Na_3$ requires 21.30 per cent of sodium.

The analysis of the salt, obtained by boiling the lactone with water and barium carbonate, led to the formula $Ba(C_{14}H_{11}O_6)_2$; the soluble zinc salt obtained in a similar way gave a percentage of zinc which agrees with the formula $Zn(C_{14}H_{11}O_6)_2$.

102. "On β -oxycellulose." By BENJAMIN SAMUEL BULL, M.A., B.Sc.

β -oxycellulose has been studied by several workers, and Cross and Bevan have prepared a trinitro-derivative. In this paper a benzoate and nitrate obtained from β -oxycellulose are described. These compounds are probably hexa-derivatives of a substance having the empirical formula $C_{16}H_{27}O_{14}$.

103. "A New Synthesis of Phloroglucinol." By DAVID S. JERDAN, B.Sc.

When finely divided sodium is dissolved in a benzene solution of ethylic acetone-di-carboxylate, and the solution is boiled for some hours, a gummy deposit is slowly formed. The whole is then shaken with water, and the aqueous solution, after separation of the benzene, is acidified with sulphuric acid. The solution becomes milky, and a granular precipitate falls after a short time. The new substance may be re-crystallised from glacial acetic acid, and then possesses a composition corresponding with the formula $C_{12}H_{10}O_7$.

This compound, when boiled with methylic alcohol containing 3 per cent of hydrogen chloride, took up a molecule of alcohol, giving a crystalline ester, $C_{13}H_{14}O_8$. The substance $C_{12}H_{10}O_7$ must therefore be a lactone. Further, on hydrolysis with baryta solution, the lactone gave carbon dioxide, alcohol, malonic acid, and phloroglucinol. The new compound is therefore a phloroglucinol derivative.

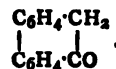
It is probably formed according to the equation—



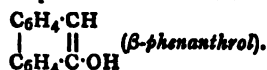
The immediate product of the reaction must of course be a sodium derivative, the lactone $C_{12}H_{10}O_7$ being formed from this on addition of sulphuric acid.

104. "Phenanthrone." By FRANCIS R. JAPP, F.R.S., and ALEXANDER FINDLAY, M.A., D.Sc.

Phenanthrone was regarded by its discoverer, Lachowicz (*J. Pr. Chem.*, 1883, [2], xxviii., 173), as a ketone of the formula—



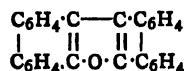
Japp and Klingemann (*Trans.*, 1893, lxxiii., 770) suggested that it might be a phenol of the formula—



In the hope of deciding between these two formulae, the present authors have prepared various derivatives of phenanthrone. The evidence, however, points in both directions. The compound reacts both in the ketonic

and in the phenolic form, although more frequently in the latter. In the great majority of its reactions it is a strict analogue of β -naphthol.

In preparing phenanthrone by the method discovered by Japp and Klingemann—reduction of phenanthraquinone with hydriodic acid—the authors find that two other substances are simultaneously formed: β -phenanthrylic oxide, $(C_{14}H_9)_2O$ (m. p. 210°), and tetraphenylenefurfuran,—

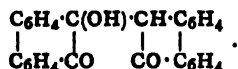


(m. p. 306°). The latter was obtained by Japp and Klingemann by the destructive distillation of monacetyl phenanthraquinol.

Phenanthrone and β -phenanthrylic oxide both yield molecular compounds with picric acid,—



(m. p. 185°) and $(C_{14}H_9)_2O \cdot 2C_6H_2(NO_2)_3OH$ (m. p. 148°). Phenanthrone, when in solution, is converted by aerial oxidation into the compound $C_{28}H_{18}O_3$ (obtained by another process by Japp and Klingemann), which crystallises in dark red laminae melting at $156-157^\circ$. This compound is broken up by acetic anhydride into phenanthrone and phenanthraquinone, the former undergoing acetylation. It may be synthesised by the direct union of phenanthrone and phenanthraquinone. The authors regard it as an aldol condensation compound of these two substances, and ascribe to it the constitution—



On boiling with fuming hydriodic acid, it is converted quantitatively into tetraphenylenefurfuran. Acetic anhydride converts phenanthrone into β -phenanthrylic acetate, $C_{14}H_9O \cdot C_2H_3O$ (m. p. $77-78^\circ$). When heated with methylic alcohol and sulphuric acid, phenanthrone yields methylic- β -phenanthrylic oxide, $C_{14}H_9O \cdot CH_3$ (m. p. $96-97^\circ$). When heated with ammoniac it yields a mixture of β -phenanthrylamine, $C_{14}H_9 \cdot NH_2$ (m. p. 130°), and β -diphenanthrylamine, $(C_{14}H_9)_2NH$ (m. p. 237°). With phenylhydrazine at 200° it interacts, eliminating water and ammonia and yielding 2':3'-diphenyleneindole (m. p. $188-189^\circ$).

105. "The Yellow Colouring Principles of various Tannin Matters." IV. By A. G. PERKIN.

Cape sumach, the leaves of the *Colpoen compressum*, is used in South Africa as a substitute for sumach (*Rhus Coriaria*) under the name of "Pruim-bast." According to H. Procter (private communication), it contains 23 per cent of a catechol tannin. Its dyeing property is due to the presence of a new glucoside, *oxyvitrin*, $C_{27}H_{30}O_{17}$, pale yellow needles, m. p. 185° , which is decomposed by acid into quercetin and glucose,—



This is not identical with viola-quercetrin (Mandelin, *Y.*, 1883, 1369), $C_{42}H_{44}O_{24}$, which exists in the *Viola tricolor florensensis*. The tannin, obtained as an orange-coloured transparent mass, is a glucoside yielding, with acid, an anhydride or phlobaphene and a sugar. By fusion with alkali, protocatechuic acid is formed. A re-examination of gambier catechu (*Ungarica Gambier*) corroborated the statement of Löwe (*Zeit. Anal. Chem.*, 1874, xii., 127) that this contains quercetin, Acacia catechu not previously examined was found to contain the same colouring matter.

The dyeing properties of a commercial sample of Venetian sumach (*R. Cotinus*) are due to myricetin and not to quercetin as stated by Löwe (*loc. cit.*). This result will be corroborated by the examination of a specially picked sample.

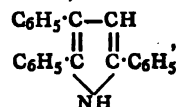
Valonia (*Quercus Egilops*), divi-divi (*Casalpina*

Coriaria), myrabolans (*Terminalia chebula*), agarobilla (*Casalpina brevifolia*), pomegranate rind (*Punica granatum*), and gall-nuts (*Quercus infectoria*), owe their tinctorial property to ellagic acid, and contain no member of the quercetin group. It is here pointed out that the plants examined hitherto contain, respectively, a tannin and colouring matter which yield on decomposition identical acids, and in some cases the same phenol.

106. "Ammonia and Phenylhydrazin Derivatives of $\alpha\beta$ -Dibenzoylcinnamene (Anhydracetophenonebenzil)." By FRANCIS R. JAPP, F.R.S., and ALFRED TINGLE, B.Sc.

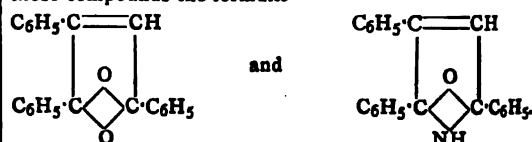
By oxidising dibenzoylcinnamenimide, $C_{22}H_{17}NO$ —the first product of the action of ammonia on dibenzoylcinnamene—with chromium trioxide, the authors have obtained a mixture of dibenzamide, benzamide, and regenerated dibenzoylcinnamene.

By reducing dibenzoylcinnamenimide with zinc dust and acetic acid in the cold, A. Smith's triphenylpyrrholone,—



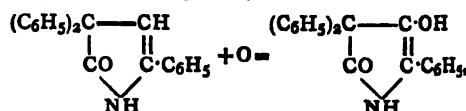
melting at $140-141^\circ$ (*Trans.*, 1890, lvii., 645), is formed.

The authors discuss the various reactions of dibenzoylcinnamene and dibenzoylcinnamenimide, and ascribe to these compounds the formulæ—



It seems to be impossible to assign to dibenzoylcinnamenimide, for example, any other formula which will account for the formation of dibenzamide during oxidation.

By the oxidation of triphenylpyrrholone—the transformation product of dibenzoylcinnamenimide under the influence of heat—with chromium trioxide, the authors have obtained a compound which they regard as triphenylhydroxy-pyrrholone (m. p. 168°),—



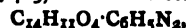
or one of its possible tautomeric forms. Heated with caustic potash, this compound evolves ammonia, and yields a mixture of benzoic and benzoic acids.

The authors have also studied the destructive distillation of the compound $C_{28}H_{22}N_2$ (m. p. about 230°), obtained by Japp and Huntly (*Trans.*, 1888, liii., 184) by the action of phenylhydrazin on dibenzoylcinnamene. They find that it yields the 1:3:4-triphenylpyrazole obtained by A. Smith (*Annalen*, 1896, cclxxxix., 332) by the destructive distillation of tetraphenyldihydro-1:2-diazine. They point out that result renders it very improbable that the compound $C_{28}H_{22}N_2$ has the constitution of an anilidotriphenylpyrrhole, ascribed to it by Japp and Klingemann (*Trans.*, 1890, lvii., 671).

107. "Derivatives of Cotoin and Phloretin." By A. G. PERKIN and H. W. MARTIN.

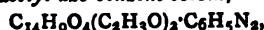
A study of the acetylation of the diazobenzene derivatives of cotoin and phloretin.

Cotoin, $C_{14}H_{12}O_4$, a constituent of coto-bark, is, according to Ciamician and Silber, a monomethyl ether of benzoylphloroglucinol, $C_6H_2(OCH_3)(OH)_2 \cdot CO \cdot C_6H_5$ (*Ber.*, 1894, xxvii., 409). Cotoinasobenzene,—



forms orange-yellow needles, m. p. $183-184^\circ$. Cotoinaso-o-toluene, $C_{14}H_{11}O_4 \cdot CH_3 \cdot C_6H_4N_2$, m. p. $203-204^\circ$,

and *cotbin-azo-p-toluene*, m. p. 207—208°, crystallise similarly. *Diacetyl-azo-benzene cotbin*,—



crystallises in scarlet needles, m. p. 155—156°. As with the maclurin compound (*Trans.*, 1897, lxxi., 186), the acetyl-groups could be determined by Liebermann's method.

Phloretin, $\text{C}_{15}\text{H}_{14}\text{O}_5$, occurs in the root bark of the apple tree as a glucoside phloridzin. According to Ciamician and Silber, it has the constitution $\text{C}_6\text{H}_2(\text{OH})_3\cdot\text{CO}\cdot\text{CH}(\text{CH}_2)_2\cdot\text{C}_6\text{H}_4\cdot\text{OH}$. *Phloretin-disazobenzene*, $\text{C}_{15}\text{H}_{12}\text{O}_5(\text{C}_6\text{H}_5\text{N}_2)_2$, red needles, m. p. 254—256°, *phloretin-disazo-o-toluene*, m. p. 250—251°, and *phloretindisazo-p-toluene*, m. p. 250—251°, closely resemble the corresponding maclurin derivatives. *Acetyl phloretindisazobenzene*, $\text{C}_{15}\text{H}_{11}\text{O}_5(\text{C}_2\text{H}_3\text{O})(\text{C}_6\text{H}_5\text{N}_2)$, forms orange-red needles melting at 217—219°. No higher acetyl derivative could be obtained. Comparing this result with those previously obtained with maclurin and phloroglucinoldisazobenzenes (*Trans.*, 1897, lxxi., 186), it would thus appear that phloretin contains only three hydroxyl groups. From Ciamician and Silber's work there appears to be no doubt, however, as to the correctness of their constitution for phloretin (*loc. cit.*). Thus, all hydroxyls in the phloroglucinol nucleus of phloretin must in diazobenzenephloretin be in the ketonic form, a peculiarity which in some way is therefore due to the influence of the phloretol group.

108. "Azobenzene Derivatives of Phloroglucinol." By A. G. PERKIN.

Though phloroglucinol is known to yield azo- and disazo-derivatives as phloroglucinol-*p*-azobenzene sulphonic acid, $\text{C}_6\text{H}_5\text{O}_3\cdot\text{N}_2\cdot\text{C}_6\text{H}_4\cdot\text{SO}_3\text{H}$ (Stebbins, *Am. Chem. Soc. J.*, 1880, ii., 240), and diazobenzenephloroglucinol, $\text{C}_6\text{H}_4\text{O}_3(\text{N}_2\cdot\text{C}_6\text{H}_5)_2$ (Weselsky and Benedikt, *Ber.*, 1879, xii., 226), no trisazo-compounds have been previously obtained, though judging from its constitution the formation of such should be expected.

Phloroglucinoltrisazobenzene, $\text{C}_6\text{H}_3\text{O}_3(\text{C}_6\text{H}_5\text{N}_2)_3$, fine needles possessing a green iridescence which do not melt below 300°, is formed by addition of diazobenzene sulphate to a solution of phloroglucinol in aqueous sodium carbonate. Its production is independent of the amount of diazobenzene sulphate employed. It contains no free hydroxyl groups, being insoluble in alkaline solutions.

Phloroglucinol-o-trisazoanisole is prepared from phloroglucinol and *o*-diazoisole in either sodium carbonate or acetate solution. No corresponding disazo-compound could be obtained in this manner. It forms maroon coloured needles melting above 300°, insoluble in alkaline solutions.

Phloroglucinol-disazobenzene-m-azonitrobenzene, obtained from phloroglucinol-disazobenzene and *m*-diazonitrobenzene, forms dull red needles, m. p. 290°.

It is proposed to study the reaction of other substituted diazobenzenes with phloroglucinol under similar conditions.

109. "The Action of Phosphorus Pentachloride on Fenchone." By J. ADDYMAN GARDNER, M.A., and G. B. COCKBURN, B.A.

Fenchone is acted on at the ordinary temperature very much more slowly than camphor, and the products of the action are different, for on pouring into water to get rid of the excess of phosphorus pentachloride and oxychloride, the authors obtained a crystalline compound of the formula $\text{C}_{10}\text{H}_{14}\text{ClPO}(\text{OH})_2$, which they name chlorofenchone-phosphoric acid, and an oil consisting of unchanged fenchone and a substance containing chlorine, probably chlorofenchone.

Chlorofenchone phosphonic acid is a white crystalline solid, melting at 196°. It is very soluble in ether, alcohol, chloroform, and benzene, but more sparingly soluble in water. It is a dibasic acid, and the sodium salt crystallises in white needle-shaped crystals. The lead, barium,

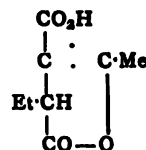
and copper salts are insoluble in water. The oil containing chlorine is at present under investigation.

110. "Ketolactonic Acid and its Homologues." By C. H. G. SPRANKLING, B.Sc.

In 1882, Young (*Trans.*, 1883, xliii., 172) observed that when β -ethylacetosuccinic ether is slowly distilled, a little alcohol is liberated, and on hydrolysis of the distillate with hydrochloric acid a crystalline acid, $\text{C}_8\text{H}_{10}\text{O}_4$, is formed in addition to α -ethyl- β -acetopropionic acid and a small quantity of ethylsuccinic acid.

The barium salt, $\text{Ba}(\text{C}_8\text{H}_9\text{O}_4)_2$, is obtained by the action of barium carbonate; a cold solution of barium hydrate gives the salt of β -ethylacetosuccinic acid, whilst at 100° barium carbonate is precipitated and the salt of α -ethyl- β -acetopropionic acid is formed.

From its composition, method of formation, and behaviour it was concluded that the crystalline acid, to which the name ketolactonic acid was given, has the constitution—



At Prof. Young's suggestion, these experiments have been repeated, and a much larger yield of the crystalline acid has been obtained by prolonged heating of the β -ethylacetosuccinic ether before hydrolysis.

The lower homologues of the acid have also been prepared in a pure state from acetosuccinic ether and β -methylacetosuccinic ether respectively, and it has been found that by prolonged heating of β -isopropylacetosuccinic ether and subsequent hydrolysis with hydrochloric acid a very small quantity of the higher homologue is formed.

It is thus shown that the crystalline acid obtained by Young is the third member of a series to which the general name ketolactonic acid may conveniently be given. It will be necessary, however, to call the lowest member of the series ketolactonic acid, the others being named methyl, ethyl, isopropylketolactonic acid.

Ketolactonic acid, $\text{C}_6\text{H}_6\text{O}_4$, does not crystallise; methylketolactonic acid, like the ethyl-compound, forms colourless crystals, m. p. 176°.

The barium salts corresponding to those derived from the ethyl-compound, were prepared from ketolactonic acid and methylketolactonic acid.

The rate of action of sodacetosuccinic ether on the brominated fatty ethereal salts, the rate of elimination of ethyl alcohol from the β -alkylacetosuccinic ethers, and the rate of hydrolysis of the ethers differ greatly in the four cases examined, the rate in general diminishing rapidly with rise of molecular weight.

The hydrolysis of the β -alkylacetosuccinic ethers may take place in two ways—(a) The acetyl-group is replaced by hydrogen and an alkylsuccinic acid is formed; (b) carbon dioxide is evolved and an α -alkyl- β -acetopropionic acid formed. With the ethers investigated, the higher the molecular weight of the alkyl-group the larger is the relative yield of the alkyl succinic acid.

PHYSICAL SOCIETY.

Ordinary Meeting, November 12th, 1897.

Mr. G. JOHNSTONE STONEY, Vice-President, in the Chair.

MR. J. ROSE-INNES read a paper on "The Isothermals of Ether."

The well-known generalisations of Boyle and Gay-Lussac with regard to the pressure, volume, and temperature relations of gases, were examined by Ramsay and

Young, who deduced the law $p = bt - a$, i. e., that pressure is a linear function of temperature, at constant volume, where b and a are functions of volume only. It yet remains to discover the form of these two functions, b and a . The author finds b and a for a large number of volumes, and from them devises an empirical formula. As a preliminary step he examines whether any single algebraical expression can represent the case, so as to determine the probability of discontinuity. For this purpose a graphic method is applied. By plotting $(aV^*)^{-1}$, against $V - t$, a curve is obtained of "cusp"-shape. The point of the cusp occurs very near critical volume; it suggests discontinuity in the slope of $(aV^*)^{-1}$. The author concludes that there is extremely rapid change of behaviour of the gas at this point. Again, it is known that the temperature at which pressure is accurately given by the laws of a perfect gas at a particular volume is constant for large volumes until critical volume is approached. The author observes that at the critical volume this temperature diminishes somewhat from its value for large volumes. These conclusions were embodied in a previous paper, and an algebraical expression for pressure in terms of temperature and volume were then given for isopentane. In the present paper the author investigates a similar formula for ether.

Prof. RAMSAY said that experimental errors might account for some of the lack of agreement between proposed formulæ and direct observation of the behaviour of gases. Isopentane was probably a better investigated body than ether, for it was simpler. Ether tended to form complex molecular groupings, but isopentane was probably a mono-molecular liquid.

Prof. PERRY did not quite agree with the author's conclusions. It was necessary to distinguish between a formula founded on a physical hypothesis and a mere empirical formula. The author had assumed that the Ramsay and Young formula was very exact; its originators did not put it forward as being infinitely exact. Probably the best test for such a formula as that under discussion would be derived from some thermo-dynamical conclusion deduced from it. The Rose-Innes formula with five constants and implying discontinuity, was to be distrusted, for there was no such thing as discontinuity in the problem. In any case, an empirical formula should have a very simple form.

Mr. ROSE-INNES admitted that a formula founded on sound hypothesis was to be preferred to empirical expressions. But mathematicians had not yet provided a hypothesis applicable to a substance whose molecular arrangement was so complicated as that of ether. Mathematicians must therefore improve their methods before working formulæ could be deduced from their hypotheses. The use of an empirical formula with five constants was justified by Kepler for the planetary orbits. Kepler used that formula with no other justification than his experience that an ellipse fitted his observations better than a circle. Similar instances might be cited from recent work on the theory of solution, and osmotics.

Mr. JOHNSTONE STONEY was disposed to look for a mathematical cause for the cusp; it was improbable that the physical change was so abrupt as that represented graphically by the author. The question might be tested by plotting the two curves $y = V - t$ and $y = aV^*$, and by observing whether these also suggested discontinuity.

Mr. W. L. WATERS then read a paper on the "*Variations in the E.M.F. of the H-form of Clark Cells with Temperature.*"

The authors, Messrs. F. S. Spiers, F. Twyman, and W. L. Waters, have investigated how nearly the true E.M.F. of Clark cells can be computed at different temperatures by applying the ordinary temperature correction. As a standard, two cells of the Muirhead type are employed. The four cells under test could be put through cycles of temperature in a special heating-bath, containing oil circulated by a centrifugal pumping-

vane. E.M.F.'s were determined by a potentiometer method, and a careful study was made of the "lag" of E.M.F. behind temperature. The results are given in the form of curves. It is shown that "lag," in the H-form of cell, is less than in the "Board of Trade" form. Under ordinary conditions, when the rate of variation of temperature is less than 2°C . per hour, by applying temperature-corrections the true E.M.F. of the H-form can be found to within a ten-thousandth of a volt. In this respect there is little to choose between the H-form and the "Muirhead" cell.

Mr. W. R. COOPER thought the authors did not express the case clearly. The E.M.F. of the "Board of Trade" cell could not, with reason, be itself stated within 1 per cent. But in some cases, when for instance cells were used differentially, greater accuracy might be required, as for example when a constant source of E.M.F. was being compared with the variations of another source; here it might be necessary to know the "lag." He would like to know with what degree of accuracy the E.M.F. of the standard cell was determined by the authors. The lag that occurred in the "Board of Trade" cell was probably due to diffusion, crystallisation, and solution.

Mr. WATERS said the E.M.F. of the standard was measured by a Kelvin balance, to one in ten-thousand.

The VICE-PRESIDENT proposed a vote of thanks to the authors, and the meeting adjourned until Nov. 26th.

THE CHEMICAL AND METALLURGICAL SOCIETY OF SOUTH AFRICA.

Meeting held August 21, 1897, at Johannesburg.

Mr. CHAS. BUTTERS, President, in the Chair.

AFTER the election of several new members, the meeting proceeded to discuss a number of amendments to the by-laws of the Society, principally with regard to the reduction of the subscription and widening the qualifications for membership. After the general business had been disposed of, a special meeting was held at which the amendments were all carried unanimously.

The meeting proceeded to discuss the President's Address.

Mr. J. R. WILLIAMS paid a well-deserved compliment to the ability of the President and the excellence of his address, but asked for an explanation of one or two points concerning the roasting and cyaniding of the ore.

Mr. CROSSE confirmed Mr. Williams's experience of having obtained poor results from extracting with cyanide, even after partial roasting; Barberton ore contained 30 to 50 per cent of pyrites, and the results from cyaniding were very poor.

After a few remarks from other gentlemen with regard to the effect of arsenic, the debate on Mr. Williams's paper on the "*Treatment of Battery Slimes*" was opened.

Mr. FRANKLIN WHITE, who spoke at some length, criticised the figures given as to the percentage efficiency of treating the slimes, and pointed out that Mr. Williams distinctly hinted that the number of tons treated was less than 6643, though that figure was used in calculating the cost out to the hundredth of a penny, and further, that by an error on the part of the accountant, the cost of working is arrived at by using the total number of tons crushed as a divisor, viz., 54,531, instead of 21,539, the actual weight of slimes; this of course, he contended, introduces a serious error.

In reply, however, Mr. WILLIAMS ridiculed Mr. White's contention, and claimed that his method of calculation was correct.

Mr. CALDECOTT pointed out that dry crushing and direct cyanide treatment is largely practised in New Zealand and America, and that there was in the Transvaal, though not on the Randt, a Company using this method at a total cost of only ten shillings per ton.

A few remarks were then made by Mr. Cross on Mr. Caldecott's paper on "*The Solution of Gold in Accumulated and Other Slimes*," the further discussion of which was postponed to the next meeting.

Meeting held September 18, 1897.

Mr. CHAS. BUTTERS, President, in the chair.

In the further discussion of Mr. Williams's paper on the "Treatment of Battery Slimes," the point was raised as to the most efficient method adopted for stirring the slimes, either by mechanical stirrers or by the introduction of air.

The PRESIDENT was of the opinion that a larger volume could be agitated at a less cost by using paddles than by air; but he thinks there is possibly a field for the younger men who adopt agitation solely by air. The time necessary for agitation depends entirely on whether the material is fresh or old, and whether it contains much reducing matter; at the City and Suburban it is as high as 18 hours, but they are not even then quite satisfied with their gold solution.

Mr. MACINTYRE, who has been treating slimes at Spitzkop for about six months, found that by increasing the number of blades in the mechanical stirrers the cone towards the centre was entirely got rid of; the time for treatment was about two days, and in a small plant about 15 tons a day were treated.

After a few remarks from one or two other gentlemen the further discussion on this paper was adjourned, and the discussion on Mr. Caldecott's paper on "*The Solution of Gold in Accumulated and Other Slimes*" was resumed.

Mr. CROSSE referred to the variation in the specific gravity of different slimes, five samples of which were as follows:—Robinson, 2.38; Meyer and Charlton, 2.59; Goldenhuis Estate, 2.31; Lancaster, 2.50; and Bonanza, 2.62. This is an important subject, as, for the purpose of calculating the amount of slimes in the liquid, the specific gravity will have to be determined before the necessary factor can be applied.

The PRESIDENT, in referring to the use of air, said he considered it as one of the most important discoveries we have had in connection with the cyanide process.

Dr. LOEY considered that the question of the succession in which oxidation takes place in pyritic ores was an open one, and will require more discussion and experimenting on before it can be decided.

This concluded the discussion, and Mr. Caldecott will reply at the next meeting.

Mr. CROSSE then read a paper entitled "*Some Notes on Assaying Ground Graphite Crucibles*." What is needed for this assay is a reagent that will give enough oxygen to burn away all the graphite, and then lose its excess of oxygen, so that a nearly neutral body is left. Mr. Crosse finds that powdered dioxide of manganese satisfies the required conditions. He mixes 10 grms. of the sample with 35 grms. of the dioxide in a H crucible and raises the temperature to a bright red heat; he then reduces the temperature and adds a mixture of—

100 grms. flux (carb. potash 2 parts, borax 1, salt 1).
50 " litharge.
2 " flour.
20 " silica.

The whole fuses together perfectly: when finished the crucible is cooled and broken open, and a soft lead button is obtained containing all the gold.

During the discussion which followed this paper the PRESIDENT introduced some notes on the by-products from the gold industry, viz., from the mills, the chlorination works, the cyanide works, and the melting room, and after thoroughly treating the subject he recommended that the attention of managers and directors should be given to these sources of income, remarking that frequently the battery is utilised as the great sewer through which these valuable products disappear.

CORRESPONDENCE.

THORIUM ACETYL-ACETONATE.

To the Editor of the Chemical News.

SIR,—In reply to "Allanite," no doubt his question will be answered by M. G. Urbain, the author of the article on "Thorium," but, if not, it will please "Allanite" to learn that acetylacetone is not ordinary acetone. Acetylacetone, $\text{CH}_3\text{—CO—CH}_2\text{—CO—CH}_3$, is prepared by either of two methods, viz.,—(1) by the action of Al_2Cl_6 upon acetyl chloride; (2) by the action of Na upon a mixture of aceto-acetic ether and "ordinary" acetone. It is a liquid with a b.p. 137° .

The works he refers to in his letter are too old, but it is described in Bernthsen, p. 238.

Hoping now he will get over his difficulty—I am, &c.,

J. A. FOSTER, A.I.C.
Assist. Admiralty Chemist.

H.M. Dockyard, Portsmouth,
November 13, 1897.

WIRE GAUZE.

To the Editor of the Chemical News.

SIR,—No doubt many of your readers, like myself, have had great trouble with their wire gauze for the tops of burners and stands. I have tried many different kinds, and find that ordinary nickel steel gauze, costing about threepence a square foot, is very satisfactory. It does not rust or perish to anything like the extent that other gauzes do.—I am, &c.,

H. L. ROBINSON.
Chemical Laboratory, Vickers, Sons, & Maxim,
Erith, Kent, Nov. 14, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Seances, de l'Academie des Sciences. Vol. cxxv., No. 18, November 2, 1897.

Preparation and Properties of Calcium, Strontium, and Barium Borides.—H. Moissan and P. Williams.—The authors arrive at the following conclusions:—The three alkaline earthy metals, calcium, barium, and strontium, yield with boron compounds of the formula B_6R , a formula identical with that of the nitrides of Curtius. These compounds are perfectly crystalline; they scratch ruby, possess a great stability, do not decompose cold water as do the carbides, and are especially destroyed by oxidising agents; they are not comparable to the alkaline-earthly carbides and silicides in composition and in properties.

On the Atomic Weights of Argon and Helium.—H. Wilde.—This paper will be inserted at some extent.

On Stannic Acids.—R. Engel.—The results of the author's researches may be summarised as follows:—Pure metastannic acid, isolated from a metastannate or from metastannyl chloride, and dried in a dry vacuum, has the composition $(\text{SnO}_2)_5 \cdot 5\text{H}_2\text{O}$ assigned to it by Fremy in his second study. It contains about 11 per cent of water (or theoretically 10.7).

The Use of Fluoresceine for the Detection of Traces of Bromine in a Saline Mixture.—H. Baubigny.

The Crystallographic Identity of Dextro-rotatory and of Levo-rotatory Asparagine.—P. Freundler.

A Study of the Transformation of Saccharine Matter into Oil in Olives.—C. Gerber.—Olives present a quotient superior to unity when the proportion of mannite decreases and that of oil increases. This quotient is due to the formation in the oil itself of more olive oil at the expense of the mannite.

MISCELLANEOUS.

Royal Institution.—Professor Oliver Lodge, F.R.S., will deliver the first of a Course of Six Christmas Lectures (specially adapted to young people) on "The Principles of the Electric Telegraph," at the Royal Institution, on December 28th. The remaining Lectures will be given on Dec. 30, 1897, and January 1, 4, 6, 8, 1898.

Imperial Institute.—The Winter Course of Lectures at the Imperial Institute will be opened on Friday, November 19th, at 9 p.m., when His Royal Highness the Prince of Wales, President of the Institute, will occupy the chair at an illustrated Lecture by Mr. F. G. Jackson, F.R.G.S., leader of the Jackson-Harmsworth Expedition, entitled "Three Years in the Arctic."

On Monday, the 22nd of November, at 8.30 p.m., Mr. E. S. Bruce, M.A., will read a paper on "Electric Balloon Signalling applied to Scientific Exploration in Arctic and Antarctic Expeditions," fully illustrated by working models and experiments, at which Major P. A. MacMahon, R.A., F.R.S., will preside.

On Monday, November 29th, at 8.30, Sir George Scott Robertson, D.C.L., will deliver an illustrated Lecture on "The Wild Kafirs of the Hindu Kush."

On Monday, December 6th, an illustrated Lecture on "The Mineral Resources of British Columbia and the Yukon" will be given by Mr. A. J. MacMillan, of Rossland, B.C., formerly British Agent for the Government of Manitoba, who has been specially supplied with specimens, &c., to illustrate this Lecture, by the Government of British Columbia. The Hon. Forbes G. Vernon, Agent-General for British Columbia, will take the chair.

On Monday, December 13th, Professor W. C. Roberts-Austen, C.B., F.R.S., will Lecture on "Canada's Medals"; the Right Hon. Lord Strathcona and Mount Royal, G.C.M.G., High Commissioner for Canada, will preside.

On Monday, December 20th, Mr. Boverton Redwood, F.R.S.E., will Lecture on "The Petroleum Sources of the British Empire."

Of the above Lectures, those on the 6th and 13th of December, by Mr. A. J. MacMillan and Professor Roberts-Austen respectively, will be open free to the public (seats being reserved for Fellows of the Institute); the others are open only to Fellows of the Imperial Institute and persons introduced by them.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Calcium Sulphate.—Will any reader kindly inform me whether there is any considerable demand for crude calcium sulphate and the approximate price per ton.—B.

Finishing Woollen Goods.—I am anxious to ascertain a simple and inexpensive mode of washing and finishing large quantities of woollen goods without them shrinking. I should therefore be much obliged if you could put me in communication with any person having this special knowledge.—G. W. WALKER, Springfield Bleach Works, Bulwell, Nottingham.

MEETINGS FOR THE WEEK.

WEDNESDAY, 24th.—Society of Arts, 8. "Progress of Metallurgy and Metal Mining in America during the Last Half Century," by Prof. James Douglas.

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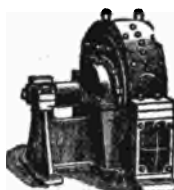


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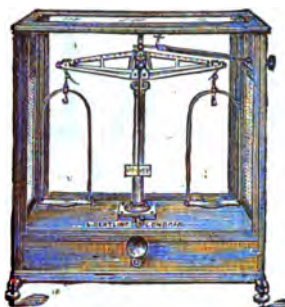
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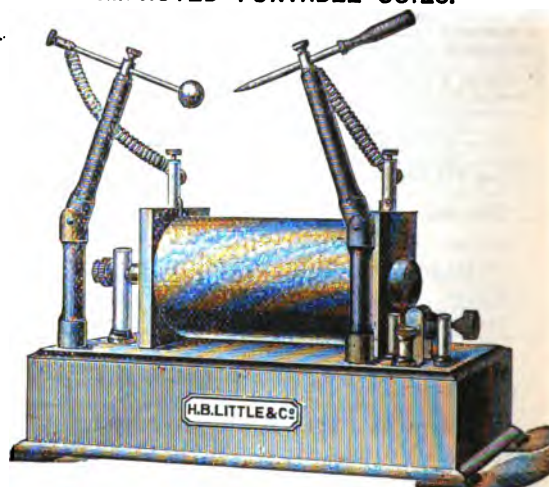
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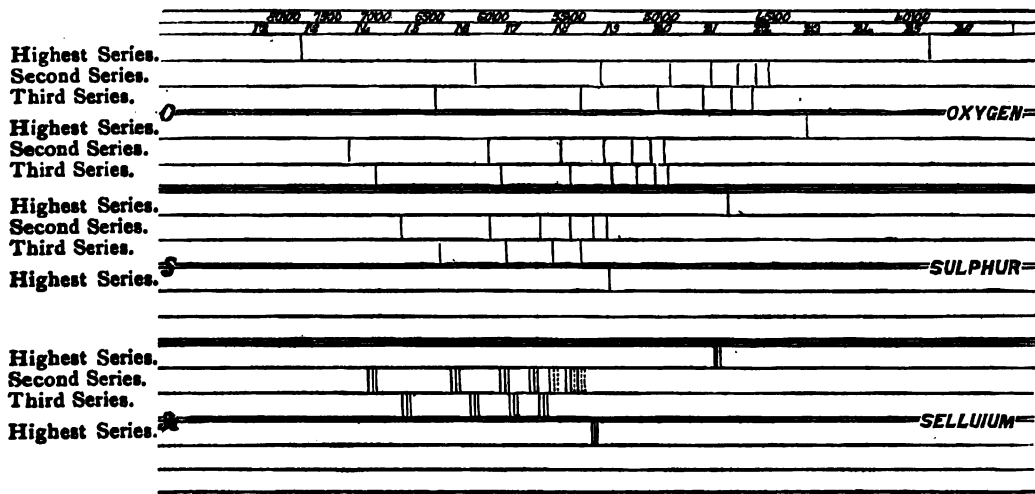
VOL. LXXVI., No. 1983.

ON THE SPECTRA OF OXYGEN, SULPHUR, AND SELENIUM.*

By C. RUNGE and F. PASCHEN.

Two years ago Prof. Pasteur and I showed that the spectrum of helium consisted of six so-called series, which may be arranged in two sets of three, each set resembling very closely the spectrum of one of the alkali metals. From this fact we drew the conclusion that helium probably consisted of two elements, one element corresponding to each of the two sets. Since then we have made an investigation of the spectrum of oxygen as it is exhibited by the electric current passing through a vacuum tube containing oxygen, when no spark gap or Leyden jar is interposed in the circuit; and we have found that this spectrum

row contrast, the second one on the right being much narrower than the one on the left. Secondly, whereas in the triplets of the second and third row the strongest line has the lowest wave-number, and the weakest line the highest, this order is reversed in the triplets of the first row. This is in perfect accordance with the spectra of the alkalis. There we have doublets instead of triplets; but we find the same two circumstances. Whereas in the so-called secondary series, corresponding here to the second and third row, the difference of wave-numbers remains the same, it grows smaller with increasing wave-number in the so-called principal series corresponding to the first row. Secondly, whereas in the secondary series the stronger line has the lower wave-number, this order is reversed in the principal series. This is connected with a law discovered by Rydberg. Rydberg showed that in the spectra of the alkalis the difference between the wave-numbers of the common limit of the two secondary series and the wave-number of the limit of the principal series is equal to the wave-number of the first member of the principal series. Now there are two limits of the secondary series, one for the stronger lines and one for the weaker lines of the doublets; but there is only one limit of the principal series. Therefore there are two differences, a , b , between the limits of the secondary series and the limit of the principal series, a corresponding to the limit of the



of oxygen closely resembles that of helium. You see a drawing of it here, together with the spectra of sulphur and selenium plotted to the scale of wave-numbers, 20, for instance, corresponding to the wave-length $1/20,000$ c.m., equal to 5000 Angström's units.

But this is only a very crude representation of what is actually seen. The drawing only gives the lines, not their intensity, which decreases with the increasing wave-number. Neither does it render the beautiful details of the lines giving an untiring sense of pleasure to the eye. The lines of the second and third row, for instance, are triplets of very characteristic appearance,—so characteristic that Piazzi Smyth, who was the first to observe some of them, very justly remarks that it is as easy to recognise one of them in the spectrum of a vacuum tube swarming with impurities as it is to pick out from among a crowd of civilians a soldier with scarlet coat and cross-belts. The first row also consists of triplets; but they differ in two very remarkable ways from the triplets of the second and third row. Whereas here the differences of wave-numbers are the same for all the triplets, the triplets of the first

stronger lines, b to that of the weaker lines. Now as the principal series overlaps the secondary series, a is larger than b , and therefore the first member of the principal series consists of a doublet with the wave-numbers a and b , the larger number a corresponding to the stronger line. Rydberg had the boldness to predict that his law would hold good for triplets, although principal series of triplets had not yet been observed; and he was right. We see his law obeyed in the spectrum of oxygen, and similarly in that of sulphur and selenium.

I cannot enter into the details of the mathematical formulæ representing the wave-numbers of each series, and proving the analogy of the three series of each set with the three series of each of the alkali metals. But what Professor Paschen and I wish to convey is this:—*There is about as much spectroscopic evidence for the duplicity of oxygen that there is for the duplicity of helium. Therefore, if on chemical reasons we are not allowed to consider oxygen as consisting of two elements, then spectroscopy does not give us any reason to believe in the duplicity of helium.*

At the same time we have investigated the spectrum of sulphur and of selenium, two bodies chemically related to oxygen, and we have found that under analogous conditions

* Read before the British Association (Section A), Toronto Meeting, 1897. See also *Wied. Ann.*, Bd. 62, 1897.

both emit a spectrum very similar to the set of the first three series. Whether the three other series also find their analogy we do not venture to contend. There is, however, a strong line in the spectrum of sulphur, as well as in that of selenium, that seems to correspond to the strong violet oxygen line.

The three series of the spectrum of sulphur and of selenium are triplets of the same build as the oxygen triplets; only they are wider, the difference of wave-numbers being, roughly speaking, proportional to the squares of the atomic weights, a law that also holds good for several other groups of chemically related elements.

The spectrum of oxygen has been partly observed by other spectroscopists. Piazzzi Smyth and A. Schuster should be named in particular; but these spectra of sulphur and selenium have now been observed for the first time. It is strange that it should be possible to find new spectra of bodies so well known as sulphur and selenium; and we can perhaps infer from this fact that many of the lines which astronomers observe in the spectra of the sun and of the stars, the origin of which is not known, may after all belong to well-known elements.

The spectrum of sulphur as a whole consists of lines of smaller wave-numbers, that is to say of slower oscillations, than that of oxygen; and the spectrum of selenium consists, again, of slower oscillations than that of sulphur. This is what we should expect from the atomic weights of the substances, the heavier atoms oscillating slower than the lighter ones.

In analysing the radiations emitted by the elements, spectroscopy can as yet give us very little insight into the nature of these complex, variable, and mysterious things that science pleases to call elements; but we think a time will come when the regular distribution of lines in what we call series will find a mechanical explanation. And that, we think, will shed a flood of light on the nature of things.

ON THE IMPURITIES OF COMMERCIAL CARBIDES OF CALCIUM.

By H. LE CHATELIER.

THE study of the impurities of carbide of calcium is interesting on account of the indications it can give as to the reciprocal affinities of certain bodies when heated to a temperature of about 2000°.

After calcium and carbon, the two most abundant elements present are silicon and iron. It is possible that definite combinations of either of these bodies may exist with each of the other three; which are those, we may ask, which are formed by preference at the temperature of solidification of calcium carbide?

The iron, which is the least abundant of the four elements under consideration, might be entirely saturated by one or the other of the remaining three. As a matter of fact it is combined exclusively with the silicon. It is detected by treating the hydrated carbide with an acid, and suspending the insoluble residue in iodide of methyl. Small crystals of silicide of iron are precipitated; these have been studied by M. Moissan; by analysis they have been proved to be SiFe_2 .

The excess of silicon combines either with the carbon or with the calcium, according to the relative proportions of these two bodies; if the quantity of carbon present is in excess of the calcium, a silicide of carbon is formed, crystallising in apparently hexagonal plates, and generally of a blue colour. It is found, with the excess of graphite, floating on the surface of the iodide of methyl. If, on the contrary, the quantity of calcium is in excess of the quantity of carbon present, a silicide of calcium is formed, which is disseminated throughout the mass of

the carbide in the form of metallic grains, having the colour and lustre of zinc. These can be isolated by rapidly quenching the carbide in a large excess of cold water, separating by levigation the coarser residues, and washing them for a few moments with a solution of acetic acid. The final residue is composed of silicide of iron, and of the coarsest grains of silicide of calcium, which have withstood the rapid washings intended to isolate them. In such a case there would be neither graphite nor silicide of carbon present, for this reason, that the calcium must be in excess to allow of the formation of silicide of calcium. In fact, the determining affinities which govern the combination of these elements are those of iron for silicon, and of calcium for carbon. These are first satisfied, and the remaining bodies combine amongst themselves in a manner varying according as one or the other is in preponderating proportion.

There seem to exist two distinct silicides of calcium; one of them is hardly attacked by nitric acid, but is, on the contrary, easily attacked by hydrochloric acid, with the formation of an insoluble yellow matter, called *silicons* by Wöhler. The other, easily attacked by nitric and acetic acids, gives, with hydrochloric acid, a deposit not yellow but white, which, like *silicons*, is soluble in potash, with an abundant disengagement of hydrogen.

In the attack of silicide of calcium we more frequently get the yellow and white compounds together. The analyses of these mixtures lead one to suspect compounds between those corresponding to $\text{Si}_2\text{O}_4\text{H}_4$ and $\text{Si}_2\text{O}_3\text{H}_4$. For example, one analysis gave—

Yellow matter	0.52 gm.
Hydrogen given off (0° and 760 m.m.) ..	630 c.c.
Silica	0.57 gm.

which corresponds exactly to the second formula.—*Bull. Soc. Chim.*, Series 3, xvii.-xviii., Nos. 16, 17.

THE COMBUSTION OF ORGANIC SUBSTANCES IN THE WET WAY.*

By I. K. PHELPS.

(Concluded from p. 247).

Method of Oxidation with Chromic Acid.

A CONCENTRATED mixture of chromic and sulphuric acids, although a much more powerful oxidiser than potassium permanganate in aqueous solutions, fails to oxidise completely many organic compounds. Thus, Cross and Higgin (*Journ. Chem. Soc.*, 1882, 113) have shown that carbohydrates are among the number of organic substances; later, Cross and Bevan find that carbohydrates and many other substances are oxidised completely to a mixture of carbon dioxide and monoxide. Messinger (*Ber.*, xxiii., 2756) has proven that carbon may be determined in organic compounds by passing the mixed products, resulting from the oxidation with chromic and sulphuric acids, through a short combustion-tube filled with granular copper oxide and heated in a furnace—all of which facts have been confirmed in my own experience.

Ludwig (*Am. Chem. Pharm.*, clxii., 47) has observed that the contact of carbon monoxide with a mixture of chromic and sulphuric acids, especially when hot, results in the oxidation of that gas to carbon dioxide. This fact suggested the idea of substituting for the apparatus described above a new form, adapted to retain the first products of oxidation in prolonged contact with the oxidising mixture. This apparatus, shown in the accompanying figure, by means of which as the sequel shows, it has been found possible to extend the availability of the oxidising mixture, is put together as follows:—A

* Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, Series 4, Vol. iv., No. 23, November, 1897.

thick-walled round bottomed flask of a litre's capacity, serving as an oxidising chamber, was closed by a rubber stopper with two perforations, through one of which passes the tube of a separating funnel of about 100 c.m.³ capacity. The tube of this funnel reached nearly to the bottom of the flask and is drawn out at the lower end. A disc of platinum foil is hung in the neck of the flask, nearly closing it, and held in place by a platinum wire passing through the foil and tucked under the rubber stopper where the funnel tube enters. The second hole of the stopper is filled by the exit tube, a glass tube of 0.7 c.m. internal diameter. This tube is expanded just above the stopper to a small bulb which serves to prevent mechanical loss of the solid contents of the flask during the boiling. This tube is joined by means of a rubber connector (provided with a screw pinchcock) to the inlet-tube of the absorption flask, which is an ordinary 500 c.m.³ round-bottom flask. This flask is also closed by a rubber stopper with two perforations, through one of which passes the inlet-tube described above and through the other the exit tube, which is also enlarged to a small bulb just above the stopper and is closed by a rubber connector and screw pinchcock. The glass ground stopper of the funnel tube is carefully cleaned and lubricated with a thick solution of metaphosphoric acid.



Instead of getting the vacuum by the water-pump, it may be gotten almost as quickly and certainly more simply by boiling water in the evolution flask and the barium hydroxide solution in the absorption flask at the same time—both flasks being connected ready for making a determination. When steam issues in good quantity from the exit tube of the absorption flask, the burner is removed from under the evolution flask and its screw pinchcock closed, and then the burner under the absorption flask and its screw pinchcock also quickly closed. The flasks are then allowed to cool.

In making a determination, the organic substance is weighed out in a counterbalanced bulb, so thin that it may be easily broken later, and made with a wide mouth for convenience in introducing the solid substance. After the substance is weighed, the mouth of the bulb is sealed by heating in a small blowpipe flame, and the tube introduced into the evolution flask, together with an amount of pure potassium dichromate, which is known to be in excess of that required to oxidise the organic substance. The flasks are connected, as already described, with an appropriate amount of barium hydroxide solution in the absorption flask and 10 c.m.³ of pure water in the evolution flask, and the vacuum obtained (as described above) by boiling both flasks, the boiling being stopped when the water in the evolution flask has decreased to 2 or 3 c.m.³. Naturally, this boiling must be so regulated as not to allow loss of the solid material in either flask. The vacuum obtained, the tube containing the organic substance is broken by shaking the flask, and 20 c.m.³ of con-

centrated sulphuric acid, previously purified from organic material by heating to the fuming point with a few crystals of potassium dichromate, are run in through the funnel tube, when reduction of the chromic acid soon becomes evident. While still hot, the acid is shaken in the flask violently, the platinum foil hung in the neck serving to protect the rubber stopper. The flask is warmed to approximately 105° C., the highest temperature to which, as shown by Cross and Bevan (*Journ. Chem. Soc.*, liii., 889), a mixture of chromic and sulphuric acid may be safely heated without the disengagement of oxygen gas. Water is then run in until the crystals of chromic anhydride have disappeared and the danger of the evolution of oxygen is passed. The solution is heated to its boiling-point, care being taken that it shall not get under pressure, which can easily be observed by opening momentarily the stopcock of the funnel tube and noting the direction of the flow of water contained in the funnel. The flask is shaken and heated alternately for five minutes—a period of time which appears to be sufficient to bring about the oxidation of the small amount of carbon monoxide originally produced. Then more water (60 to 70 c.m.³) is introduced through the funnel and the stopcock between the boiling and absorption flasks opened, when the carbon dioxide enters the absorption flask, which is kept cool and shaken as before. The contents of the evolution flask are then heated to boiling and a slow current of air, freed from carbon dioxide by passage through potash bulbs, allowed to enter through the funnel tube to keep the liquid from undue bumping. The boiling is continued for fifteen minutes, after which the excess of barium hydroxide is determined iodometrically, and thus the carbon dioxide present estimated as before. Table IV. shows results obtained by the treatment of crystallised ammonium oxalate and cane-sugar, re-crystallised from dilute alcoholic solution, in this manner.

TABLE IV.					
Substance taken.	BaO ₂ H ₂ taken.	CO ₂ found.	CO ₂ found.	CO ₂ calculated.	Error on CO ₂ .
Grm.	Grm.	Grm.	Grm.	Grm.	Grm.
<i>Analysis of Ammonium Oxalate.</i>					
1. 0.5009	1.3534	0.1469	0.3097	0.3101	0.0004—
2. 0.5006	1.3400	0.1308	0.3103	0.3099	0.0004+
3. 0.5005	1.3400	0.1343	0.3094	0.3098	0.0004—
4. 1.0002	2.5460	0.1347	0.6188	0.6192	0.0004—
5. 1.0010	2.5192	0.1094	0.6185	0.6197	0.0012—

Analysis of Cane-sugar.

1. 0.2001	1.3926	0.1905	0.3085	0.3088	0.0003—
2. 0.2000	1.3926	0.1936	0.3077	0.3086	0.0009—
3. 0.2001	1.3926	0.1857	0.3097	0.3088	0.0009+
4. 0.2014	1.3400	0.1279	0.3111	0.3108	0.0003+

The results are evidently very satisfactory.

The Determination of the Oxygen required to Oxidise an Organic Substance.

Several different methods have been proposed for estimating the oxygen present in organic substances, depending in general upon the determination of the oxygen which must be supplied to burn the substance to a known amount of carbon dioxide and water—thus discovering by difference the oxygen originally contained in the substance. Lavoisier is said to have measured directly the oxygen used in burning organic substances; Gay-Lussac and Thénard determined the oxygen used by measuring the amount of potassium chlorate reduced by burning the organic compound; Baumhauer (*Ann. Chem. Pharm.*, xc., 228) determined the oxygen used by measuring the volume of oxygen entering the combustion furnace and subtracting the measure of the gas coming from the combustion tube, which was set up according to the well-known method for determining carbon and hydrogen; Stromeyer (*Ann. Chem. Pharm.*, cxvii., 247) determined the amount of copper reduced by the ignition

of the substance in copper oxide; Ladenburg (*Ann. Chem. Pharm.*, cxxv., 1) oxidised the substance by heating in a sealed tube with a known amount of iodic acid, determining at the end of the operation the amount of iodic acid left; Mitscherlich (*Pogg. Ann.*, cxxx., 536) has estimated the oxygen in organic substances directly by decomposing the substance by ignition in a stream of chlorine gas, estimating the oxygen content by determining the resulting carbon dioxide and monoxide.

As it has been shown in the work described that carbon may be determined in organic substances by oxidation with chromic and sulphuric acids without the evolution of oxygen gas, it would seem that the determination of the oxygen in the substance might be effected by determining the amount of chromic acid used in the operation, taking into consideration the products of combustion. This can be readily accomplished by taking a weighed amount of pure potassium dichromate as the oxidising agent and determining, at the end of the operation, the amount of chromic acid left by treatment of the residue with hydrochloric acid, absorption of the chlorine evolved in an alkaline arsenite of known strength, and titration of the excess of that substance with decinormal iodine solution.

To test the accuracy of the determination of chromic acid under these conditions of analysis, weighed portions of pure fused potassium dichromate were introduced into a Voit flask, whose outlet tube was sealed to the inlet tube of a Drexel wash bottle, the outlet of which, in turn, was sealed to a Will and Varrentrapp absorption apparatus. An amount of hydrochloric acid, more than enough to completely reduce the chromate (15 to 40 c.m.³ of the strongest acid), was added with 20 c.m.³ of strong sulphuric acid, and the total volume made up to 120–140 c.m.³ of liquid. The sulphuric acid used here was purified from carbonaceous matter (as in the carbon determination above) by heating with a few crystals of potassium dichromate, the excess of which was reduced by holding the acid at a fuming point for about two hours, when a portion diluted with water gave no colour with potassium iodide and starch paste. Pure arsenious oxide, in amount slightly in excess of that required to take up the oxygen to be given up by the chromate, was dissolved by the aid of heat in a solution of pure sodium hydroxide, taken in such quantity as to more than neutralise the arsenious acid and the hydrochloric acid used to reduce chromate, and this solution was introduced into the

Drexel wash bottle. The flask was then connected with the wash bottle, using a thick solution of metaphosphoric acid to lute the joint between the flask and its stopper. The absorption apparatus was charged with a dilute solution of sodium hydroxide. Carbon dioxide was generated in a Kipp generator by the action of hydrochloric acid on marble and purified from reducing matter by bubbling through a strong solution of iodine in potassium iodide, and finally washed with a solution of potassium iodide alone. A slow stream of this purified carbon dioxide was allowed to enter the inlet tube of the Voit flask, the contents of which were then boiled. When a concentration to a volume of 30 to 40 c.m.³ was reached, the boiling was discontinued, and, after cooling and disconnecting the flask, the contents of the receiver were made acid with sulphuric acid and then alkaline with acid potassium carbonate, when the excess of arsenite was determined by titration with decinormal iodine solution. Sometimes during the reduction of the chromic acid, the red fumes of the chlorochromic anhydride volatilised to the receiver; but since the chromic acid thus produced is reduced later by the arsenite (Browning, *Am. Journ. Sci.*, 1896, vol. i., 35), this transfer is of no account in the working of the process. The following results were thus obtained:—

TABLE V.

	K ₂ Cr ₂ O ₇ taken. Grms.	As ₂ O ₃ taken. Grms.	As ₂ O ₃ found. Grm.	K ₂ Cr ₂ O ₇ found. Grms.	Error on K ₂ Cr ₂ O ₇ . Grm.
1.	5.0002	5.1025	0.1144	4.9447	0.0555—
2.	5.0018	5.0799	0.0526	4.9849	0.0169—
3.	5.0005	5.0801	0.0582	4.9782	0.0223—
4.	5.0013	5.0706	0.0908	4.9365	0.0648—

The cause of the error shown in these experiments was traced finally to too great concentration of the sulphuric

TABLE VI.

	K ₂ Cr ₂ O ₇ taken. Grms.	As ₂ O ₃ taken. Grms.	As ₂ O ₃ found. Grm.	K ₂ Cr ₂ O ₇ found. Grms.	Error on K ₂ Cr ₂ O ₇ . Grm.
1.	1.0004	1.0500	0.0398	1.0014	0.0014+
2.	1.0007	1.0531	0.0437	1.0006	0.0001—
3.	2.0013	2.0501	0.0299	2.0026	0.0013+
4.	2.0037	2.0727	0.0502	2.0049	0.0012+
5.	5.0020	5.1002	0.0495	5.0068	0.0048+
6.	5.0037	5.1018	0.0513	5.0066	0.0029+

TABLE VII.

	Substance taken. Grm.	CO ₂ found. Grm.	Error on CO ₂ . Grm.	K ₂ Cr ₂ O ₇ taken. Grm.	As ₂ O ₃ taken. Grm.	As ₂ O ₃ found. Grm.	Oxygen used. Grm.	Oxygen required by theory. Grm.	Error on oxygen. Grm.
<i>Analysis of Ammonium Oxalate.</i>									
1.	1.0122	0.6265	0.0001—	2.0009	1.3002	0.0900	0.1160	0.1139	0.0021+
2.	1.0019	0.6212	0.0010+	2.0002	1.3517	0.0440	0.1147	0.1128	0.0019+
<i>Analysis of Phthalic Acid.</i>									
1.	0.1002	0.2138	0.0014+	2.0012	1.2004	0.0814	0.1456	0.1448	0.0008+
2.	0.1093	0.2324	0.0007+	2.0000	1.1031	0.0634	0.1582	0.1580	0.0002+
<i>Analysis of Cane-sugar.</i>									
1.	0.2025	0.3117	0.0008—	3.0000	1.7002	0.0796	0.2275	0.2273	0.0002+
2.	0.4012	0.6166	0.0024—	5.0000	2.3022	0.0366	0.4495	0.4502	0.0007—
<i>Analysis of Paper.</i>									
1.	0.3034	0.4932	0.0010—	3.5015	1.4017	0.0879	0.3589	0.3598	0.0005—
2.	0.4523	0.7334	0.0033—	5.0035	1.8000	0.0710	0.5368	0.5358	0.0010+
<i>Analysis of Tartar Emetic.</i>									
1.	0.5057	0.2671	0.0009—	2.5018	1.7000	0.0766	0.1459	0.1462	0.0003—
2.	1.0099	0.5321	0.0030—	3.5003	1.7520	0.0198	0.2911	0.2919	0.0008—
<i>Analysis of Barium Formate.</i>									
1.	1.0079	0.3906	0.0006+	3.0026	2.2002	0.0496	0.1423	0.1422	0.0001+
2.	1.5014	0.5814	0.0005+	3.0010	1.8080	0.0890	0.2118	0.2118	0.0000±

acid in the process. When the boiling begins the chromate is reduced gradually and if the evaporation of the water is pushed too rapidly, the sulphuric acid may reach a strength at which it begins to cause the reduction of the chromic acid with the evolution of oxygen instead of chlorine.

The obvious remedy is to conduct the boiling operation more slowly. It was found that, if from five to six hours time was taken for the proper concentration of the contents of the Voit flask, the presence of the sulphuric acid worked no harm, as will be seen from the following results. Experiments 1 and 5 were made with 5 c.m.³ of sulphuric acid present and the others with 20 c.m.³ as used before. (Table VI.).

In applying this method to the determination of oxygen used in the oxidation of an organic substance, the carbon determination was made as already described, the amount of water used being such as to leave 60 to 80 c.m.³ of liquid in the boiling flask after the carbon dioxide had been driven to the absorption flask by boiling. This liquid was then washed into the Voit flask and the chromic acid remaining determined by a second distillation (this time with hydrochloric acid) in the manner described above. In each of the experiments recorded in Table VII., 20 c.m.³ of purified sulphuric acid were used in the carbon determination, and 35 c.m.³ of hydrochloric acid (sp. gr. 1.2) in the chromic acid determination. The ammonium oxalate used was the pure crystallised salt; the phthalic acid was re-crystallised from its water solution and dried for a short time over sulphuric acid; the cane-sugar was selected crystals of rock candy, re-crystallised from dilute alcoholic solution and dried for a long time over sulphuric acid; the paper was ashless filter-paper, dried to a constant weight over sulphuric acid; the tartar emetic was re-crystallised from water solution and air dried; the barium formate was prepared by treating formic acid with an excess of pure barium carbonate, filtering hot, and allowing the product to crystallise.

From these results, it will be seen that the process works with accuracy upon a great variety of organic substances. It was found impossible, however, to determine the elements in bodies which are at the same time volatile and hard to oxidise; for instance, ether oxidises easily to acetic acid but difficultly beyond that stage; although the liquid acid is oxidised vigorously by chromic and sulphuric acids, the gaseous acid is hardly attacked at the temperature used; naphthalene was also found to be volatilised, and hence not attacked, to such an extent as to render its determination by this process valueless.

In conclusion, the author wishes to express his thanks to Prof. F. A. Gooch for many helpful suggestions.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, November 4th, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

Messrs. Harold W. Harrie, W. R. Lang, W. H. Barlow, and A. V. C. Fenby were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Ernest George Annis, Health Office, Town Hall, Huddersfield; William Ball, 54, Stretton Road, Leicester; Richard Oxley Burland, J.P., Poolstock House, Wigan; Alexander McLean Cameron, Daylesford, Victoria; Owen Aly Clark, 12, Abbey Gate Street, Bury St. Edmunds; Alexander Clarkson, 2, Waveney Crescent, Ballymena, Ireland; Frank Collingridge, B.Sc., Kenmore, Shepherd's Hill, Highgate, N.; James Murray Crofts, B.A., Richleigh, Gloucester; David Crole, Primrose Studios, Wellington Square, Chelsea, S.W.; John Daniell, Council of Education Laboratory, Johannesburg, S.A.R.; Andrew

James Dixon, Dapto, N.S.W.; Robert Hamilton, 11, Ibrox Place, Glasgow; John Harger, B.Sc., Ph.D., The Nook, St. James's Mount, Liverpool; Charles Kelly, Oakmere, Hawarden, Cheshire; Tom Lemmey, B.A., Wellington College, Berks; James Scott MacLaurin, D.Sc., Mount Eden, Auckland, N.Z.; Allen Macmullen, 82, James Street, Dublin; Edward Masters, The Aloes, Hinckley Road, Leicester; John A. Mathews, 4, First Place, Brooklyn, N.Y.; Philip George Gregory Moon, 129, Rosary Road, Thorpe, Norwich; Joseph John Mooney, 34, Easter Road, Edinburgh; James Charles Philip, B.Sc., Ph.D., 16A, Merton Road, Victoria Road, Kensington, W.; Alexander Ferguson Reid, Stair Bridge, Stair, Ayrshire; Ernest Henry Roberts, Hollydale, All-farthing Lane, Wandsworth, S.W.; Edward Sydney Simpson, 34, Pier Street, Perth, West Australia; Robert Francis Woodsmith, 89, Bartholomew Close, E.C.; Frederick William Steel, Tamunua, Navua River, Fiji; Michael Edmund Stephens, Avenue House, Finchley, N.; George Stubbs, Arnaide, Hertford Road, East Finchley, N.; Edward Howard Tripp, Ph.D., Kent House, Blackheath Hill, S.E.; John Scriven Turner, 20, Bury Street, Bloomsbury, W.C.; Framjee Khursedjee Viccajee, Hyderabad, Deccan, India; Percy John Vinter, Wesley College, Sheffield; Arthur James White, Whinsfield, Barrow-in-Furness.

Sir WILLIAM CROOKES then took the Chair, and of the following papers those marked * were read:—

*III. "On the Properties of Liquid Fluorine." By Professors MOISSAN and DEWAR.

The nearest approach to the properties of the mythical alkalest or universal solvent of the alchemist is to be met with in fluorine. The transparent vessels in which it can be manipulated have to be made of some fluoride like fluor-spar, and such vessels are equally difficult to construct and ill-adapted for chemical manipulation. Modern research has, however, revealed the fact that the most powerful chemical affinities are completely suspended by allowing substances to come into contact at very low temperatures, and it appeared possible that even fluorine, which has the most powerful chemical activity of all the elements, might be manipulated in glass vessels under such conditions.

In a paper communicated to this Society entitled "The Liquefaction of Air and Research at Low Temperatures" (*Proc.*, 1895, xi., 221), speaking of fluorine, the author remarked, "This is the only widely distributed element that has not been liquefied. Some years ago, Wallach and Hensler pointed out that an examination of the boiling-points of substituted halogen organic compounds led to the conclusion that, although the atomic weight of fluorine is nineteen times that of hydrogen, yet it must in the free state approach hydrogen in volatility. This view is confirmed by the specific refractive index which Gladstone showed was rather lower than hydrogen. If the chemical energy of fluorine at low temperatures is abolished like that of other active substances, then some kind of glass, or other transparent material not so brittle as calcium fluoride, could be employed in the form of a tube, and its liquefaction achieved by the use of hydrogen as a cooling agent."

The inference that fluorine approached hydrogen in volatility was deduced by Wallach and Hensler from a consideration of the boiling-points of the fluorine derivatives of the benzene series.

The following table—

Benzene.	Bolling point.	Differ- ence.	I.	Toluene.	Bolling point.	Differ- ence.
C ₆ H ₆	80°	5°	}	C ₆ H ₅ CH ₃	111°	5°
C ₆ H ₅ Fl	85°			p-C ₆ H ₄ Fl-CH ₃	116°	
C ₆ H ₅ Cl	132°	47°	}	p-C ₆ H ₄ Cl-CH ₃	160°	44°

Aniline.		Benzene.	
$C_6H_5NH_2$.. 183°	} 4° 43°	C_6H_6 80°	} 8° 84°
$p-C_6H_4F \cdot NH_2$ 187°		$p-C_6H_4F_2$.. 88°	
$p-C_6H_4Cl \cdot NH_2$ 230°		$p-C_6H_4Cl_2$.. 172°	

shows that the substitution of 1 atom of hydrogen in these compounds by fluorine only causes an increase of the boiling-point of from 4° to 5° , whereas chlorine causes an increase of from 45° to 50° . Such a relatively large ratio as 1 to 10 in the increment of boiling-points suggests a great difference in the volatility of the elements fluorine and chlorine in the free state. A further examination of the properties of fluorine compounds, however, showed that the volatility of fluorine was not likely to approach that of hydrogen. This will be apparent from the following table. —

II.			
Methane. (absolute).	Boiling point Difference.	Ethane. (absolute).	Boiling point Difference.
CH_4 .. 110°	} 90°	C_2H_6 .. 184°	} 58°
CH_3F 200°		C_2H_5F .. 242°	
CH_3Cl 250°	} 50°	C_2H_5Cl 286°	} 44°
CH_4 .. 110°		C_2H_4 .. 110°	
CF_4 .. 259°	$149^\circ = 4 \times 37^\circ$	CCl_4 .. 351°	$241^\circ = 4 \times 60^\circ$
Aldehyde. B. p. (C.)		Benzaldehyde. B. p. (C.)	
CH_3COH 21°	} -11°	C_6H_5COH 179°	} -18°
CH_3COF 10°		C_6H_5COF 161°	
CH_3COCl 51°	41°	C_6H_5COCl 199°	38°

where it is seen that the substitution of hydrogen by fluorine in methane and ethane raises the boiling-point by 90° and 60° respectively, and that the ratio of the increments of boiling-point in corresponding fluorine and chlorine compounds is now not greater than 1 : 2. The boiling-point of methyl fluoride was calculated from the critical point and vapour pressure of this substance as recorded by Professor Collie (*Trans.*, 1889, iv., 110). It will be noted as a curious fact that the substitution of fluorine in the aldehyde radicle causes a lowering of the boiling-point and not an increase, and that the difference in boiling-point between the chlorine and fluorine substitution body in either series is always between 40° and 50° . These considerations induced the hope that liquid air might give the command of a sufficiently low temperature for the liquefaction of fluorine, and that glass vessels might be used to collect the liquid. This view was supported by a consideration of the melting-points of the halogens and the corresponding critical points deduced by following the suggestions of Clarke (*Am. Chem. Soc. T.*, 1896, xviii., 618) as to these relations. Thus the absolute melting-points of chlorine, bromine, and iodine are respectively 171° , 267° and 388° , and, assuming the same mean difference in melting-point extended to fluorine, then its melting-point would be 64° absolute. Now the critical points of chlorine and bromine are about 2½ times the absolute melting-points, thus giving 149° absolute, or -125° , as the probable critical point of fluorine. This critical value is only a few degrees lower than oxygen, and from this calculation the authors were entitled to assume that the position of fluorine as regards volatility would be somewhere between that of oxygen and nitrogen.

The following research was conducted in the Chemical Laboratory of the Royal Institution, to which Professor

Moissan brought the apparatus for the production of gaseous fluorine with which his name will always be identified, and the authors had the invaluable assistance of Messrs. Lebeau, Lennox, and Heath in the conduct of the experiments.

Fluorine was prepared by the electrolysis of potassium fluoride in solution in anhydrous hydrofluoric acid. The fluorine gas was freed from vapours of hydrofluoric acid by being passed through a serpentine of platinum cooled by a mixture of solid carbonic acid and alcohol. Two platinum tubes filled with perfectly dry sodium fluoride completed the purification.

The apparatus used for liquefying the gas consisted of a small cylinder of thin glass, to the upper part of which was fused a platinum tube. This latter contained in its axis another smaller tube, likewise of platinum. The gas to be liquefied enters by the annular space, passes through the glass envelope, and escapes through the small inner tube. The glass envelope was fused to the platinum tube by which the fluorine was supplied.

The glass cylinder being cooled down to the temperature of boiling liquid oxygen (-183°), the current of fluorine gas passed through the bulb without becoming liquid. At this low temperature, however, the gas has lost its chemical activity, and no longer attacks the glass.

On lowering the temperature of the liquid oxygen by exhaustion, a yellow liquid is seen collecting in the glass envelope, while gas no longer escapes from the apparatus. At this moment the tube by which the gas had been escaping is stopped, so as to prevent air from entering and liquefying, and the glass bulb soon becomes full of a clear yellow liquid, possessed of great mobility. The colour of this liquid is the same as that of fluorine gas when examined in a stratum 1 metre thick. Fluorine thus becomes liquid, according to this experiment, at about -185° .

When the bulb containing the liquid fluorine is lifted above the surface of the liquid oxygen, the yellow liquid begins to boil with an abundant disengagement of gas, having all the energetic reactions of fluorine.

Silicon, boron, carbon, sulphur, phosphorus, and reduced iron, cooled in liquid oxygen and then placed in an atmosphere of fluorine, did not become incandescent. At this low temperature, fluorine did not displace iodine from iodides. However, its chemical energy is still sufficiently great to decompose benzene or oil of turpentine with incandescence. It would thus seem that the powerful affinity of fluorine for hydrogen is the last to disappear. The authors have noticed on some occasions that a current of fluorine gas passed into liquid oxygen gives a flocculent precipitate of a white colour, which quickly settles to the bottom. If this mixture is shaken and thrown on a filter, the substance can be collected. It possesses the curious property of deflagrating with violence as soon as the temperature rises.

A new apparatus (Fig. 1) was constructed similarly to that already described (that is to say, a glass bulb, *x*, fused to a platinum tube, *A*, which contained another similar smaller tube, *B*), but having each of the platinum tubes, *A* and *C*, fitted with a screw valve, in such a manner that at any moment communication—either with the outer air or with the current of fluorine—could be interrupted. This little apparatus was placed in a cylindrical glass vacuum vessel containing liquid oxygen, connected with a vacuum pump and manometer.

On repeating the former experiment with freshly prepared liquid air, instead of oxygen, fluorine easily becomes liquid at -190° C. With liquid oxygen as refrigerant, the liquefaction of fluorine takes place at a temperature corresponding to the evaporation of the oxygen under a pressure of 437 m.m. of mercury.

From these two experiments it results that the boiling-point of fluorine is very close to -187° . This number is identical with Olszewski's boiling-point of argon, so that this seems to be the first example of two gaseous elements boiling at the same temperature. It is a justifiable infer-

ence from the boiling-point that the critical point must be about -120° , and thus, in all probability, the critical pressure is about 40 atmospheres, or less than half that of the critical pressure of chlorine, which is 84 atmospheres. This would make the critical constant for fluorine 4 as contrasted with chlorine, which has the value 5.

The following table gives the boiling-point of the halogens:—

	Absolute temperature.	Difference.
Fluorine	87°	153°
Chlorine	240°	
Bromine	337°	97°
Iodine	460°	

When the little glass bulb was three-quarters full of liquid fluorine, both the valves were closed, and then a good air-pump caused the liquid oxygen serving as refrigerant to boil rapidly at a pressure of 2.5 c.m. Under these conditions a temperature of -210° is reached, yet the fluorine did not show any sign of solidification, but retained its characteristic mobility. In future experiments it will be interesting to try the rapid ebullition of the liquid fluorine itself. During the repetition of this experiment a slight accident occurred. The screw of one of the valves becoming worn, allowed air to leak into the exhausted bulb. This air was immediately liquefied, and in a few moments two distinct layers of liquid were seen; the upper colourless layer, consisted of liquid air; the lower one, of a pale yellow colour, being fluorine.

To prevent the possible ingress of any air, the fluorine was introduced in its liquid state into a glass tube, the end of which was then sealed before the blowpipe. The sealed tube, containing the liquid fluorine, was kept for a long time at -210° by the rapid evaporation of a large quantity of liquid air, but it gave no trace of a solid body.

To determine the density of liquid fluorine, it was brought into contact with a number of bodies whose density is known, comparing their behaviour at the same time in liquid oxygen, which has about the same boiling-point and density. By taking groups of bodies whose densities are very close to each other, it is easy to see which sink and which float in the liquid. This well-known though indirect method was the most suitable for these delicate experiments. The authors first satisfied themselves that the fluorine had no action on the materials used. To effect this, a crystal of ammonium thiocyanate (density = 1.31) was placed in a glass tube surrounded with boiling liquid air to the bottom of the tube, a current of fluorine gas was introduced by means of a platinum jet. The fluorine was rapidly liquefied, and the ammonium thiocyanate was not attacked. The same experiment was repeated with a fragment of ebonite ($d = 1.15$), of caoutchouc ($d = 0.99$), of wood ($d = 0.96$), of amber ($d = 1.11$), and of methyl oxalate ($d = 1.15$). It is of importance, in the experiments just mentioned, that the various materials used should be first kept at a temperature of -190° for some little time before coming in contact with liquid fluorine.

In one of the experiments a piece of caoutchouc, having been insufficiently cooled, took fire on the surface of the liquid, and burnt completely away with a brilliant flame without leaving any residue of carbon. The piece of caoutchouc ran about the surface of the liquid like sodium on water, giving a very intense light.

The density experiment was carried out in the following manner:—In a glass tube closed at one end, and of which the lower part had been slightly drawn out, fragments of the five substances just mentioned were placed. The tube was then plunged to a third of its length into boiling liquid air. When it was all reduced to a temperature of about -190° the fluorine gas was carefully intro-

duced. This soon liquefied, and the wood and the caoutchouc floated easily on the surface of the pale yellow liquid. On the other hand, the methyl oxalate and ebonite remained at the bottom, while the amber rose and fell in the liquid, appearing to be of the same density. The apparatus was shaken several times, and the quantity of liquid fluorine increased, but the results were the same.

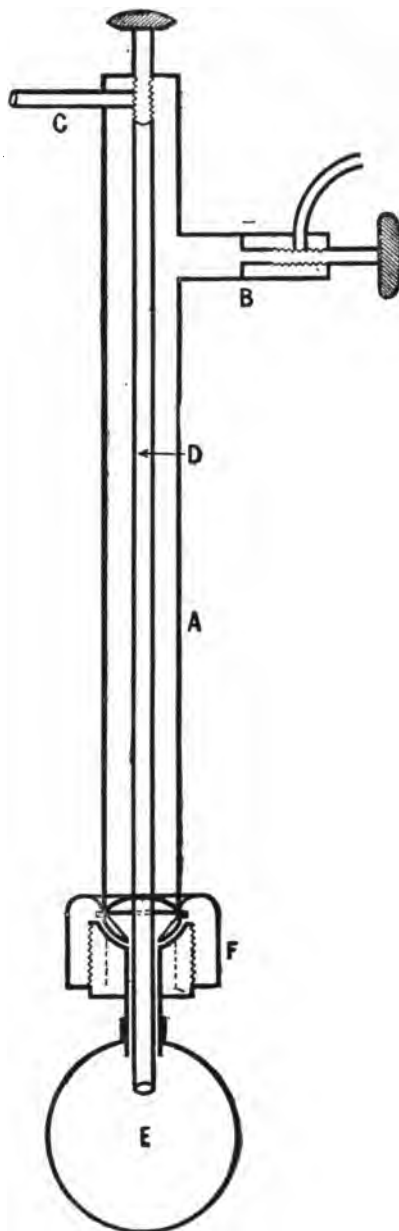


FIG. 1.

The authors thus arrive at the conclusion from these experiments that the density of liquid fluorine is about 1.14. Another point which appears to be of interest is the following:—The fragment of amber floating in the fluorine was very difficult to distinguish, which would seem to indicate that the index of refraction of liquid fluorine is in any case greater than that of liquid air or

oxygen, although it is not likely to be so high as that of amber itself.

Fluorine was liquefied in a thick-walled glass tube which had been previously graduated, and the tube sealed. On cooling the tube and its contents to -210° , a contraction of $\frac{1}{4}$ th in the volume of the liquid fluorine took place. A similar tube was left alone in a vacuum vessel full of liquid air. An hour and a half afterwards, the tube still being in liquid air, the fluorine had not changed in appearance. But shortly afterwards, when the air had all evaporated, a violent detonation occurred; the sealed tube and the double beaker in which it had been placed were smashed and reduced to powder.

Different samples of liquid fluorine examined with the spectroscope through a thickness of about $\frac{1}{4}$ c.m. showed no specific absorption-bands in the visible spectrum.

Liquid fluorine placed between the poles of a powerful electro-magnet does not show any magnetic phenomena. These experiments are the more decisive, as comparative ones with liquid oxygen were made at the same time.

The capillary constant of fluorine is smaller than that of liquid oxygen. A capillary tube, plunged successively in fluorine, oxygen, alcohol, and water, gave the following figures:—

Height of liquid fluorine	3.5 m.m.
" " oxygen	5.0 "
" alcohol	14.0 "
" water	22.0 "

Liquid fluorine placed in a glass tube surrounded with liquid air (temperature about -190° C.) had a slow current of hydrogen gas directed on to its surface by means of a fine platinum jet. There was immediate combustion with the production of flame. The experiment was repeated by dipping the platinum jet well below the surface of the liquid. At this temperature complete combination still took place, with a considerable evolution of light and heat.

Oil of turpentine, in the solid state, is attacked by liquid fluorine. To perform this experiment a little oil of turpentine was placed at the bottom of a glass tube surrounded with boiling liquid air. As soon as a small quantity of fluorine was liquefied on the surface of the solid, combination took place with explosive force, a brilliant flash of light, and deposition of carbon. After each explosion the current of fluorine gas was kept up slowly, a fresh quantity of liquid fluorine was formed, and the detonations succeeded each other at intervals of from six to seven minutes. Finally, after a longer interval of about nine minutes, the quantity of fluorine formed was sufficient to cause, at the moment of the reaction, the complete destruction of the apparatus. In several of these experiments a little liquid fluorine accidentally fell on the floor; the wood instantly took fire.

The action of liquid oxygen has been studied with more care, since the authors observed that by passing a current of fluorine through liquid oxygen a detonating powder could be produced.

If a current of fluorine is directed to the surface of liquid oxygen in a glass tube, the temperature being about -190° , the fluorine dissolves in all proportions, imparting a yellowish colour, and giving the liquid a graded tint from the upper to the lower part; the bottom of the tube is hardly coloured. If, on the contrary, the fluorine gas is introduced at the bottom of the liquid oxygen, the yellow colour is produced at the bottom and diffuses slowly to the upper layers.

This phenomenon indicates that the densities of liquid fluorine and oxygen are very near each other. When the temperature of the mixture of liquid oxygen and fluorine is allowed to rise slowly, the oxygen evaporates first. The liquid becomes more and more concentrated as regards fluorine, and finally the latter begins to boil in its turn. In fact, at the commencement of this boiling the gas coming off will light a match which has only a red-hot point, and will not make lamp-black or silicon red-hot;

but, on the other hand, the gas coming off at the end of the experiment will instantly cause these two latter bodies to burst into flame. When the glass bulb is completely empty and its temperature is rising, a distinct disengagement of heat is suddenly noticed, and the interior of the glass loses its polish. This rise in temperature is due to the fluorine gas attacking the glass. In this experiment, using perfectly dry oxygen, no precipitate is produced. If, on the contrary, oxygen is used which has been some hours in contact with the air, the detonating substance mentioned in previous experiments is produced.

The body which is produced by the action of fluorine on oxygen containing in suspension minute crystals of ice seems to be a hydrate of fluorine, decomposing, with detonation, by a simple rise of temperature. This view must, however, be taken as conjecture, until the real composition is ascertained. A small quantity of water at the bottom of a glass tube being cooled down to -190° , liquid fluorine formed on the surface of the ice as a mobile liquid without showing any chemical action, and evaporated on the temperature rising. As soon as the apparatus became warmer the remaining gaseous fluorine attacked the ice with great energy, causing a strong smell of ozone.

A globule of mercury was treated in the same way as the water described above. The surface remaining very brilliant, the liquid fluorine surrounded it without causing any diminution of metallic lustre. On allowing the temperature to rise, the fluorine began to boil, and the liquid disappeared completely, without any attack of the mercury. The experiments seem to warrant the following conclusions.

Fluorine gas is easily liquefied at the temperature of boiling atmospheric air. The boiling-point of liquid fluorine is -187° . It is soluble in all proportions in liquid oxygen and in liquid air. It does not solidify at -210° . Its density is 1.14, its capillarity is less than that of liquid oxygen; it has no absorption spectrum, and it is not magnetic.

Finally, at -190° it has no action on dry oxygen, water, or mercury, but it reacts, with incandescence, on hydrogen and oil of turpentine. Future experiments must decide whether cooling below -200° can suspend the powerful chemical action of liquid fluorine on hydrogen and hydrocarbons.

One of the most important questions for future investigation is the specific refractive and dispersive indices of the fluid. Davy, in his paper on the substances produced in different chemical processes on fluor-spar (*Phil. Trans.*, 1813, 278), says, "Dr. Wollaston has found that the fluoric combinations have very low powers of refracting light, and particularly the pure fluoric acid; so that the refracting powers of fluorine will probably be found lower than those of any other substance, and it appears to possess higher acidifying and saturating powers than either oxygen or chlorine."

Gladstone has shown that the specific atomic refraction of the combined element does not exceed 0.9, taking the Lorentz formula, and that the atomic dispersion diminishes instead of increasing for short wave-lengths. Further, he found that the other halogen substitution compounds gave atomic refractions nearly agreeing with the same substances in the free state. It has been found that liquid gases give the same atomic refraction as the gaseous body, so that the refractive index of liquid fluorine may be at once deduced provided it behaves like chlorine, bromine, or iodine. Taking 0.9 as the atomic refraction, the value would be, according to the Gladstone formula, 1.054, and the Lorentz 1.081. Both values are far lower than those of liquid oxygen or air, 1.226 and 1.205 respectively. The general appearance of the liquid and the experiment with amber described above lead to the conclusion that liquid fluorine must have a refractive index much higher than that calculated. If the refractive index is as great as 1.41, then the atomic refraction (Lorentz) will be 4.13; but if it is only about 1.192, then the atomic

refraction will be 2. On both assumptions the atomic refraction of liquid fluorine is much greater than the value 0.9 found by Gladstone. Should the smaller value 2 turn out to be the correct one, then the inference might be fairly drawn that the critical constant was also about 3, or nearly the value for oxygen. This view would make the critical pressure of fluorine about the same as that of oxygen, or 50 atmospheres. From this it would follow that, unlike chlorine, bromine, and iodine, which have the same atomic refraction in combination and in the free state, fluorine has a different value in the one state as compared to the other. In this respect it would appear to resemble oxygen, whose atomic refraction in combination may be only three-fourths of what it is in the free state. This view is confirmed by an examination of the atomic volume of fluorine. The other members of the halogen series have approximately the same atomic volume in combination as in the free state. Now, the atomic volume of fluorine in fluorobenzene is 11.5, or about half the atomic volume of chlorine, or taking chlorobenzene as standard, with chlorine as 22.7, then the atomic volume would be 10. The value for the free element appears to be 16.6, and the number deduced from liquid hydrofluoric acid about 15. Many metallic fluorides have relatively small atomic volumes. Thus the fluorides of cadmium, lithium, calcium, magnesium, and aluminium have an atomic volume just about half of that of the corresponding chloride. This difference is, however, easily explained if fluorine in the combined state has only half the atomic volume of chlorine. Dr. Thorpe's value for the atomic volume of fluorine, deduced from a study of the chloride and fluoride of arsenic, is 9.2, or free fluorine at its boiling-point ought to have a density of 2, provided it behaved like the other halogens. This density for the free element is much too high, the experimental value being about 1.14. Such changes in atomic volume again suggest a resemblance with oxygen, and would lead to the inference that the refractive constants must also differ in the free and combined states. These interesting problems must, however, be left for future investigation.

(To be continued).

EDINBURGH UNIVERSITY CHEMICAL SOCIETY.

Monday, November 15th, 1897.

THIS meeting was held in the Chemistry Tutorial Classroom.

The President, Professor CRUM BROWN, delivered an address on "*The Chemical Work of Pasteur.*"

The President gave a short account of Pasteur's life, with an outline of his chemical and biological work, treating with more detail the discovery of left tartaric acid, and of the relation between crystalline and optical enantiomorphism.

With reference to Pasteur's views as to the connection between life and asymmetry, he said, "We often hear surprise expressed that Pasteur should have continued to hold that asymmetric molecules cannot be produced without the aid of living organisms, after he had himself shown that Perkin and Duppas's acid is racemic acid capable of yielding right and left tartaric acid. A careful study of what Pasteur actually said and wrote shows that this surprise is founded on a misunderstanding. Pasteur certainly believed at the time when he delivered his lectures to the Chemical Society of Paris (1860) that not only asymmetric substances, such as tartaric acid or glucose, but also racemoids, such as racemic acid, required the active presence of living things for their production. But in the note in the *Annales de Chimie et de Physique* in which he announces his discovery that Perkin and Duppas's acid is racemic acid, we see that he had already modified his view, and allowed that racemoids could be

produced by purely laboratory processes. And in 1874 and 1875, in communications to the Academy, he states his mature opinion in perfectly clear language. We may or may not agree with him, but his notions on the subject are not in contradiction to any observed fact."

As to the origin of the asymmetry in living things—as shown, for instance, in the fermentation of right but not of left tartaric acid by *Penicillium glaucum*—the speaker suggested that there may be some analogy between this and the development of asymmetrical habits in ourselves, such as the habit of following a definite order in putting on our stockings, shoes, &c.

On concluding his address Prof. Crum Brown was, on the motion of Dr. Macdonald, awarded a hearty vote of thanks for his very interesting address.

CORRESPONDENCE.

REFORM OF CHEMICAL AND PHYSICAL CALCULATIONS.

IN the CHEMICAL NEWS for 6th of August last (vol. lxxvi., p. 70) we published a review of a book having the above title. Mr. C. J. T. Hanssen, the author of the book, objected to some expressions used in the review, considering we had held him up to ridicule. The editor at once wrote to Mr. Hanssen assuring him that no such impression was intended, nor could it be fairly gathered from the tone of the review, which on the whole was distinctly favourable to the work. Our explanation not being considered satisfactory, and wishing to be scrupulously accurate in our statements and just to the author of the book, we think it will be better to let Mr. Hanssen speak for himself in the following letter, which we publish without further comment.

MATT., vii., 2.—"With what judgment ye judge, ye shall be judged."

3 Valdemarsgade,
Copenhagen, V., 20th November, 1897.

Professor Sir William Crookes, F.R.S.,
7, Kensington Park Gardens,
London, W.

DEAR SIR,

I received your esteemed and friendly letter of the 13th inst., and day after day I have read it and compared it with your review of my book in the CHEMICAL NEWS, but I regret to say I cannot in the review discover that you recognise *the* merit or *any* merit in my book. The first 35 lines are merely quotations from the book; then follows *your judgment*: "weird and forbidding aspect" of vulgar fractions; "a certain amount of fascination" in author's idea, but impossible to adopt it, gradual or universal; it would only increase the existing confusion!

A more sweeping condemnation of my book I can hardly conceive; and such a judgment, from a first-rate scientific authority, of course kills my book, and *me* as an author on scientific objects, unless *you*—in a distinct and prominent form, with your own signature—publish another review of my book in the CHEMICAL NEWS. Your friendly private letter, and the prefix Dr., which I am not entitled to, cannot mend the harm and damage done by the public condemnation.

If you had known my book, which, to judge from your letter, you do not, I am sure you would have written the review in a different style; but as matters now stand, I expect of you, as a gentleman, the satisfaction mentioned.

If you kindly will refer to the book, you will find that I by no means wish to do away with decimals, but freely use them myself where an approximately exact result is sufficient, but that I use vulgar fractions in the deter-

mination of *exact standard values*, which only in few cases can be found by decimals and by logarithms.

It was my aim to calculate standard values *exact*; approximate values, with unreasonable long tails of decimals, we have plenty of in the text-books of all nations, and *they* are the cause of the confusion which I wish to reform.

If we take Lord Rayleigh's determination of the weight of 1 cbm. of oxygen (latitude of London) = 1.42952 kg., the weight of hydrogen = exactly $\frac{1}{16}$ thereof, nitrogen = 14, carbon = 12 times the weight of H, then—

1 cbm.	Kg.	Kg.	Cbm.
H weighs	0.089345 = 1, and 1 is	1.089345 = 11.12945	
O "	0.42952 = 16, " "	1.42952 = 0.69953	
N "	1.250830 = 14, " "	1.25083 = 0.79496	
C "	1.072140 = 12, " "	1.07214 = 0.92745	
Argon "	1.786900 = 20, " "	1.78690 = 0.55647	
Helium	0.357380 = 4, " "	1.035738 = 2.78236	

These are calculated from Lord Rayleigh's standard for oxygen, as exact as can be done by decimals, but they are only approximate. Volume $\times$ weight is *not* = 1, as it should be.

In my system the conversion of weight to volume and *vice versa* is done without calculation, and the relation between both is always correct. The standard values (for latitude $41^{\circ} 10'$) corresponding to those above are expressed thus (page 3 § 9):—

1 cbm.	Kg.	And 1 kg.—	Cbm.	Cbm.
H weighs	5/56 = 1, H	= 56/5 =	11.2	
O "	80/56 = 16, O	= 56/10 = 7/10 = 0.7		
N "	70/56 = 14, N	= 56/70 = 4/5 = 0.8		
C "	60/56 = 12, C	= 56/60 = 14/15 = 0.9333		
Argon "	100/56 = 20, Argon	= 56/100 = 14/25 = 0.56		
Helium "	20/56 = 4, Helium	= 56/20 = 14/5 = 2.8		

In the Dowson gas calculations, quoted with so much emphasis, you have overlooked that—

$$240 \times 867/140 = \frac{12 \times 867}{7}$$

(not so dreadful after all), and that this line belongs to the calculation of calors. per cbm.; and anyone looking over the next line may see at a glance that—

$$64/185 \times 4335 = \frac{64 \times 867}{37}$$

another very plain calculation. From § 25 the calors. per cbm. and per kg. may further be found by a very plain method, absolute exact.

In Table XXIII. the calors per cbm. and per kg. given for various gases. They are found, per kg., by multiplying 4335 cal. $\times$ ratios in col. 2; and per cbm. by multiplying 61924 = 43350/7 calors. $\times$ ratios in col. 6. Where, for mixed gases, the ratios are given in decimals, the results vary a trifle from the exact figures in cols. 1 and 5; the ratios in vulgar fractions for simple gases and chemical compounds give the exact values.

I think the chapters on evaporation and combustion are well worth your attention.

As the scientific world has adopted such complicated systems as the "Natural," the M.G.S., and the F.G.S., I think my plain arithmetic must be a release from the toil with Dynes, 1/745 h.-p. = 10⁷ erg., &c., &c. After swallowing the camel, they cannot be afraid of the gnat.

I hope soon to be informed that this matter will be settled in a friendly manner, and remain

Very truly yours,

C. J. T. HANSEN, C.E.

NOTE.—*Erratum*: Table XXIII., col. 7, 3 lines from bottom, instead of 0.171 please read 0.240.

MOLECULES AND LIQUEFACTION HEATS.

To the Editor of the Chemical News.

SIR,—After reading the interesting paper by Noel Deerr (CHEMICAL NEWS, vol. lxxvi., p. 234) I cannot refrain from sending you an account of a thermal relation which I discovered a year or two ago, and which shows no less constancy than any of those which he refers to, while it has the important characteristic of being grounded on theoretical considerations. In fact, it was an *a priori* consideration of theory that led me to search for and to discover the relation.

Assume, then, in accordance with van 't Hoff's theory of solutions, that the molecules of a liquid have the same translational kinetic energy as they would have if they belonged to a gas of the same temperature. The specific heat of a liquid will consist of three parts:—Firstly, the heat required to increase the kinetic translational energy of the individual molecules; secondly, the heat required to increase the internal energy of the molecules, which in the case of monatomic molecules is nil; and thirdly, the heat required to overcome intermolecular attraction.

The first of these will be identical with the gaseous specific heat at constant volume. Now, when the liquid is changed into a solid the energy arising from this cause no longer exists in the translational condition, since the molecule becomes fixed in position; that energy will therefore be liberated as heat. The energy arising from the third cause possibly does not undergo any great change in amount, unless in solidifying the volume of the liquid greatly changes. The energy due to the second cause I neglect, in order to deal with monatomic molecules alone.

If these assumptions are sufficiently correct, it must follow that the heat set free in passing from the liquid to the solid state will, in the case of a monatomic molecule, be equal to the translational kinetic energy of a gas molecule at that temperature. Thus—

The molecular liquefaction-heat of a monatomic liquid is equal to the total kinetic energy of a monatomic gas molecule at the same temperature.

This for each grm.-molecule can easily be calculated, as is well known. Taking t the absolute temperature, it is equal to about $3t$ calories.

Doubtless the molecules in the liquid state are to some extent polymerised. Raoult's investigations on solvents seem to indicate that the ratio of the theoretical number of grm.-molecules is to the true number as 1 : 0.9 or as 1.11 : 1, this having a rough generality.

Hence the law just laid down with regard to liquefaction heats will be incorrect to this extent (if we neglect the internal changes in the heat of the few polymerised molecules). Since it is true, by a rough generalisation, that the theoretical number of molecules in the liquid is to the real number as 1.11 to 1, it will follow that the gaseous molecular kinetic energy calculated at the temperature of liquefaction will be greater than the true heat of liquefaction by the same ratio.

But since Raoult's number was derived from liquids remote from their solidifying-point, we expect greater polymerisation at that point, and therefore a somewhat larger ratio. We need not expect absolute uniformity between different liquids, but a ratio less than unity is inexplicable on this theory, except on the assumption of a great internal heat in molecules polymerised.

To show that these points are satisfactorily borne out, I give a table which I calculated some time ago, with the addition of six elements whose liquefaction heat was at that time unknown to me; I now take that constant from Deerr's paper.

The column headed M gives the molecular weight of the liquid ($O = 16$). We are here confined to liquid *metals*, which are the only elements that have any probability of possessing a monatomic molecule in the gaseous (or liquid) state. M.-P. is the melting-point in absolute

degrees; L, the heat of liquefaction per unit mass; K, the gaseous kinetic energy calculated per unit mass at the same temperature. The last column is the ratio between these two.

	M.	M.-P.	L.	K.
Silver, Ag = 107.9	1227°	26.7	33.72	1.27
Cadmium, Cd = 112.1	588°	13.66	15.52	1.14
Mercury, Hg = 200.4	233°	2.82	3.45	1.22
Zinc, Zn = 65.5	685°	28.1	31.01	1.10
Platinum, Pt = 194.8	2048°	27.2	31.24	1.15
Lead, Pb = 206.9	605°	5.37	8.69	1.60
Palladium, Pd = 106	1773°	36.2	49.6	1.37

The following are after Deerr's numbers:—

Sodium, Na = 23	365°	32.7	47.48	1.45
Potassium, K = 39	335°	15.7	25.6	1.63
Copper, Cu = 63.3	1470°	43.0	55.3	1.29
Thallium, Tl = 204	560°	5.1	8.18	1.58
Aluminium, Al = 27	1150°	100.0	127.4	1.27
Gold, Au = 197	1330°	16.3	20.2	1.24

The following, which I calculated before, I found not to fulfil the law:—

Tin, Sn = 118.1	503°	13.62	12.63	0.93
Bismuth, Bi = 208	533°	12.64	7.6	0.603
Gallium, Ga = 69.9	303°	19.11	12.85	0.673

The first two have non-metallic tendencies, and their non-metallic relatives are allotropic.

On seeing Deerr's constants, I proceeded at once to make use of them in the calculation, and in no case was I disappointed. The result was six new metals well within the law. On the other hand, if we apply the principle to liquids of known polyatomic vapour or gas, we meet at once, as might be expected from theory, with enormous and disproportionate divergencies; this is in itself a confirmation. For example:—

	M.	M.-P.	L.	K.
Bromine, Br = 79.9	266°	16.2	4.98	0.303
Iodine, I = 126.9	386°	11.7	4.56	0.390

Higher molecules show still greater divergence. Still, it seems likely that an examination of such molecules (particularly those of similar structure) in the light of this theory would be fraught with important information respecting internal specific heats; and, indeed, information of a yet more far-reaching character.

I observe that Deerr mentions Crompton as giving a formula for a constant which involves atomic weight; probably it is similar to the above, or derived from it.—I am, &c.

P. J. BEVERIDGE, M.A., B.Sc.

St. Ann's, St. Helens, Nov. 15, 1897.

THE LATE VICTOR MEYER.

To the Editor of the Chemical News.

SIR,—There appears to be a strong desire among many of the British students who worked under the late Prof. Victor Meyer to give expression to the feelings of gratitude and admiration with which they remember him, by raising some form of memorial to be placed in the Heidelberg Lecture Theatre.

It has therefore been decided to call a general meeting of Prof. Meyer's British students, to be held in Manchester, on Saturday, December 11th, at 5 p.m. Prof. H. B. Dixon, F.R.S., has kindly placed the Organic Lecture Theatre of the Owens College at our disposal.

All past students of the late Victor Meyer, whether they worked with him in Zürich, Göttingen, or Heidelberg, are earnestly requested to be present.

I shall be pleased to receive suggestions from any who may be unable to attend, in order that they may be laid before the meeting.—I am, &c.,

J. J. SUDBOROUGH.

University College, Nottingham,
November 23, 1897.

ESTIMATION OF NICKEL IN STEEL.

To the Editor of the Chemical News.

SIR,—In the CHEMICAL NEWS (lxvii., p. 248) you publish an electrolytic method of estimating nickel in nickel steel. I do not think that any electrolytic method is likely to supersede the xanthate process of Messrs. Andrews and Campbell, described in the Abstracts J. C. S. (Part II., 1895, p. 421).

I have used it many times, always with good results.

No figures appear to be given in support of Mr. Durn's method, where the percentage of nickel is very low.—I am, &c.,

H. L. ROBINSON.

Chemical Laboratory, Vickers, Sons, and Maxim,
Erith, Kent, Nov. 23, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxv., No. 19, November 8, 1897.

Interpretation applicable to Faraday's Phenomena and Zeemann's Phenomena.—Henri Becquerel.

A Study of the Oysters of Cette as regards Pathogenic Microbia.—A. Sabatier, A. Ducamp, and J. M. Petit.—A pathological memoir.

Variation of Energy in Isothermic Transformations. On Electric Energy.—H. Pellet.

Dissemination of the X Rays.—A. Buguet.—The use of protective screens, which is not indefensible for short exposures before tubes of little penetrating power, becomes necessary in prolonged exposures. In medical applications they also render it possible to obtain more detailed proofs by shorter exposures.

On the Molecular Volumes and the Densities of Gases in general at any Temperature and at Mean Pressures.—A. Leduc.—A mainly mathematical paper.

On some Novel Spectral Lines of Oxygen and Thallium.—H. Wilde.—On a comparison of the wavelengths of the lines it is found that none of them coincide with those of argon. Observations on the new lines of thallium 6955, 6560, gives the author occasion to again call attention to the spectroscopic study of thallium by Stas, in which he declared that the electric spectrum of this metal consists of a simple green line incapable of being split up like the spectrum of the flame. As the eminent Belgian chemist used in this research a current derived from a series of only 30 Bunsen elements the red line in the arc spectrum escaped his observation. To resolve the second line it is necessary for the current to have the intensity of 100 volts or 50 Bunsen elements. The red line 6560 in the arc-spectrum has been observed in all specimens of thallium and its compounds which have been submitted to examination.

The Use of Fluoresceine for the Detection of Traces of Bromine in a Saline Mixture.—H. Baubigny.—The author, in concert with P. Rivals, has devised a procedure for the decomposition of the bromides, founded on the action of a mixture of permanganate and a soluble salt of copper. He has sought for a practical means of deciding if this decomposition is complete at a given moment. The fluoresceine paper is obtained very easily. Fluoresceine is prepared by heating for three hours the desired proportions of orthophthalic acid, and of resorcin, to 190 to 200°; it is purified, and then treated with pure acetic acid at 40°–30° per cent. Into the filtered solution the paper is plunged, and allowed to dry. To use this paper it is moistened, when the least trace of bromine gives a distinct rose colour. Organic matter must be excluded.—*Comptes Rendus*, cxv., No. 18.

MEETINGS FOR THE WEEK.

MONDAY, 29th.—Society of Arts, 8. (Cantor Lectures). "Gutta Serena," by Eugene F. A. Obach, Ph.D., F.C.S.
Royal Institution, 8.30. "The Wild Kaffre of the Hindu Kush," by Sir George Scott Robertson, D.C.L.

WEDNESDAY, Dec. 1st.—Society of Arts, 8. "The American Bicycle—The Theory and Practice of its Making," by Prof. Leonard Waldo, D.Sc.

THURSDAY, 2nd.—Chemical, 8. Ballot for the Election of Fellows.
"On Collie's Space-Formula for Benzene," by F. E. Matthews, Ph.D.

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Full particulars are given in the book of Regulations for Admission to the Institute, which may be obtained from Messrs. Blundell, Taylor, & Co., 173, Upper Thames Street, London, E.C., price One Shilling.

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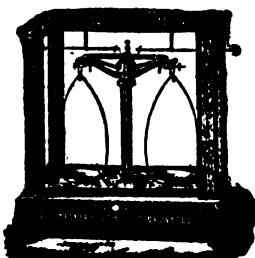
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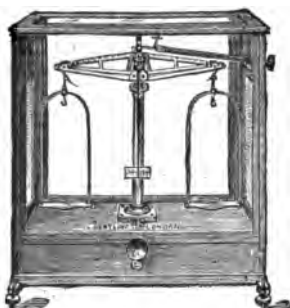
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RESULTS OF EXPERIMENTS WITH THE CHEAVIN FILTER.

By Dr. T. L. PHIPSON,

formerly of the University of Brussels and the Laboratoire de Chimie Pratique, Paris.

THIS cylindrical filter is made of porcelain (unglazed biscuit) like that known as the Pasteur-Chamberland, of which it is a modification, and in some respects an improvement. The object of this filter is to remove bacteria from water used for drinking; and it is applicable to other liquids for the same purpose.

It has been known for some time that these porcelain biscuit filters, when properly made and devoid of flaws, will remove microbes of all kinds from water which passes through them, and this has been proved in the present case by a number of experiments carried on in my laboratory during the summer months of the present year. But the passage of water through such a medium is, of course, very slow.

The yield may be increased, however, in three manners:—

1. By pressure; exerted, for instance, by a large cistern at the top of the house upon the filter in the basement.
2. By employing, with or without extra pressure, a certain number of cylinders instead of only one.
3. By producing a partial vacuum in the filter by means of a small hand pump, which pulls the water through the filter into the receiving vessel.

In the first case the cylinder is connected with a branch of the supply pipe at the basement; a single tube will thus yield each day sufficient water for drinking to supply a small family. When a larger supply is required, a certain number of the filters are joined together in a special cylindrical receptacle, and all act together.

In the third case, the use of a small hand pump, worked evenly and not too vigorously, will, by producing a partial vacuum in the receiving vessel and the porcelain cylinder, give a much more rapid yield of water also devoid of microbes. When the porcelain cylinder is placed thus in a vessel of stagnant water swarming with the "green matter of Priestley," consisting of extremely minute unicellular algae and various kinds of microbes, a few ounces may be pulled through in a few minutes, and the filtrate thus produced may be exposed to sunlight for weeks without the slightest green matter appearing, whilst ordinary river water exposed by its side as a "witness" becomes quite green.

In other experiments the water was filtered by means of the little hand pump into sterilised beef-tea and other media, with similar results.

With wines, spirits, beer, vinegar, solutions of sugar, solutions of salts, &c., the effects of the Cheavin filter are well worth attention. Cloudy wine is rendered bright without affecting its other properties. Cider, beer, and even oils can be thus rendered pure in a very short time.

When slime or mucilage has collected on the surface of the cylinders, they can be cleaned by brushing in boiling water, or by passing each tube slowly through a large naked flame. Being light and delicate in construction, these filters require some care in handling to avoid injury. At the old Fulham Potteries, where they are made in large quantities, a considerable number are broken up as unfit for use after testing. They are equally efficient for filtering air and depriving it of the microbes in suspension.

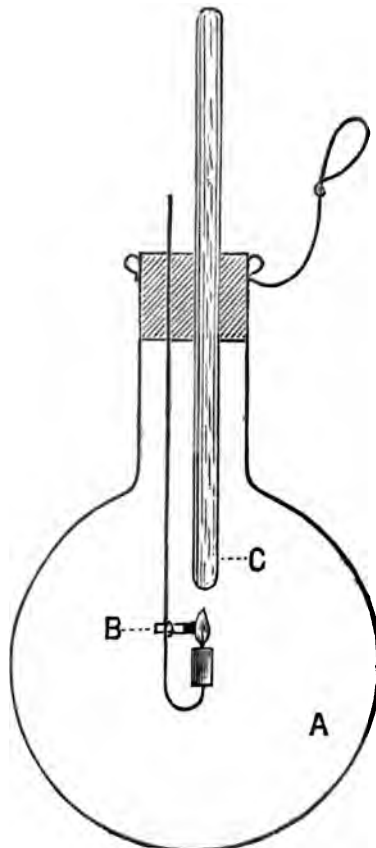
Casa Mia, Putney, S.W.

LECTURE EXPERIMENT.

By W. FRENCH.

I HAVE found the following experiment showing the effect of burning a candle in air to work extremely well, and it possesses the advantage of being very simple. It is a modification of an experiment well known, and figured in "Perkin and Lean," and dispenses with the use of a battery, which in a small laboratory is not always handy.

The round bottom flask, A (about 1 litre), is fitted with an indiarubber stopper through which passes an iron rod having a small candle attached. Just above the candle is



wired a small "match end," B, so that the head nearly touches the wick of the candle. C is a greased glass rod passing easily through the stopper. The indiarubber stopper can be wired down. The whole apparatus is counterpoised. The lower portion of the glass rod is heated, the cork replaced and wired in; the glass rod is now momentarily pushed down until it touches the match, which readily ignites the candle. When the candle ceases to burn re-weigh.

Obituary. — On November 5th C. W. Blomstrand, Professor of Mineralogy and Inorganic Chemistry at the University of Lund, closed his laborious and successful career in his 71st year.—*Chemiker Zeitung*.

Appointment.—Dr. F. Stanley Kipping, F.R.S., Lecturer and Assistant in the Chemical Research Laboratory of the Central Technical College, has been appointed to the Professorship of Chemistry in University College, Nottingham.

and it has been generally recognised that twelve hours at least was necessary. Some chemists have maintained that filtration could be proceeded with much sooner; I, in my experiments on this particular point, have met with some anomalies, of which the following is a good example:—

From a solution of a natural phosphate, freed from silica, I took nine equal volumes of 50 c.c. each (corresponding to $\frac{1}{3}$ gram. of the phosphate). To each of these samples I added, as usual, 25 c.c. of citrate of ammonia at 100 grms. of citric acid per litre, 60 c.c. of ammonia at 22°, and 20 c.c. of ammonio-magnesian chloride at 20 grms. of magnesia per litre.

The washings were effected with 45 c.c. of water containing one-third of its volume of ammonia at 22°, making a total volume of 200 c.c.

After agitation and the formation of the precipitate, the solutions were let stand for varying times. The results obtained are given in the following table:—

No.	Time of standing.	Weight of precipitate.	Remarks.
I.	1 hour	0.2329	Filtrate cloudy.
II.	2 hours	0.2338	Filtrate slightly cloudy.
III.	4 "	0.2350	Clear.
IV.	8 "	0.2351	"
V.	16 "	0.2345	"
VI.	32 "	0.2345	"
VII.	64 "	0.2342	"
VIII.	128 "	0.2338	Loss probable.
IX.	256 "	0.2343	"

It is seen at once that the weight of the calcined precipitate first of all increases for eight hours, then slowly declines to a definite limit in about sixteen hours (Experiment No. VIII. would appear to be vitiated by some accident). The above proves the occurrence of two superposed phenomena, which may be, for example:—

1. The precipitation of the phosphoric acid which takes place at once, more or less, in the state of tri-magnesian phosphate and is complete in about eight hours.
2. The much slower transformation of the tri-magnesian phosphate, at first formed as ammonio-magnesian phosphate, which takes at least sixteen hours.

These results have been confirmed by a large number of isolated experiments—which have, as a matter of fact, been the cause of the present systematic research. Two examples are here given:—

	I.	II.
Standing for 4 to 6 hours..	0.2176 grm.	0.2175 grm.
Standing for 16 to 22 hours	0.2163 "	0.2166 "

The excess in weight of the precipitate collected between four and six hours may therefore exceed that which has stood for sixteen to twenty-two hours by at least 1 m.grm. However, these facts being susceptible of different interpretations,—different, in fact, to that which has been given above,—it becomes necessary to look for a definite answer, and to primarily enquire into other experiments showing similar variations.

III.—Mechanical Precipitation.

It has been well known for some years, and even adopted officially in Belgium, that continuous shaking for a quarter of an hour or twenty minutes of the solution to which the precipitant has been added is of great advantage. This has been called the *citro-mechanical* method.

The comparative examination I have made of this method leads me to the conclusion that it gives exactly the same results, in weight of the calcined precipitate, as in letting stand for four to six hours; that is to say, an excess of about 1 m.grm. above the results obtained after sixteen to twenty hours. This conclusion has been arrived at from a large number of estimations carried on by the two methods simultaneously.

Is there really an excess in the case of rapid precipita-

tions, or does the long standing bring about a loss? What has been written up to now only shows that there is a decided difference between the two methods.

IV.—On the Influence of Dilution.

Two experiments were made with equal volumes of the same solution. The quantities of citrate and chloride of magnesium used were the same in each case; but in the second the total volume was doubled by the addition of water, but keeping the proportion of free ammonia constant. The following table gives the conditions and the results of the two experiments:—

	I.	II.
Solution of phosphate	50 c.c.	50 c.c.
Citrate of ammonia	25 "	25 "
Ammonia at 22°	60 "	120 "
Chloride of magnesia	20 "	20 "
Water	—	95 "
Wash-waters ($\frac{1}{3}$ ammonia at 22°)	40 "	80 "
Total volume	195 "	390 "

Weight of calcined precipitate.. 0.2336 grm. 0.2353 grm.

Contrary to what might be expected, the greater dilution causes an augmentation in the weight of the precipitate collected.

This systematic experiment was suggested by a certain number of results obtained in the ordinary course which had led to the same conclusions; the fact now appears to be beyond doubt.

Here, again, several explanations are possible. Either the nitrate of ammonia exercises a solvent action on the ammonio-magnesian phosphate,—an action which is the more marked as the solutions are the more concentrated, —or there remains in the precipitate tri-magnesian phosphate or phosphate of lime, proportionally greater as the solutions are more dilute.

V.—Does the Excess come from Lime?

One might suppose that in the first moments of precipitation lime, in the state of phosphate, would be carried down; its further transformation being only very gradually achieved. I was the more inclined to this belief, inasmuch as I had just come across a body which did not appear to have been previously noticed, the *ammonio calcic phosphate*.

This compound is obtained under exactly the same conditions as the ammonio-magnesian phosphate in the presence of citric acid in a strongly ammoniacal solution. It is fairly soluble in ammoniacal water, and is formed more easily at a low temperature. The precipitate is composed of small brilliant crystals, sufficiently large to be recognised by the naked eye. Its composition, according to an analysis I have made, is calculated absolutely on that of ammonio-magnesian phosphate. In spite of its relative solubility, it does not appear impossible, by reason of isomorphism, that this phosphate might be at first carried down with the ammonio-magnesian phosphate, and then slowly decomposed. I have searched—and searched in vain—to see if there is any trace of lime in the precipitates showing an excess; but there is none, and this result is beyond doubt; for to produce an excess of 1 m.grm. it would be necessary for 3 m.grms. of magnesia to be replaced by 4 m.grms. of lime—a quantity which could not fail to be detected. It is therefore necessary to put on one side the hypothesis of the presence of lime in ammonio-magnesian phosphate.

VI.

With the object of finding out the reason for these slight divergencies I carried out another series of experiments which I will now proceed to describe.

Three equal volumes of a similar solution, each corresponding to 1.25 gram. of the natural phosphate, were pre-

precipitated with proportional quantities of the usual reagents under the following conditions:—

- I. Let stand for twenty-one hours.
- II. Let stand for three hours and a half.
- III. Mechanical precipitation in twenty minutes.

After thorough washing, the three precipitates were redissolved in hydrochloric acid, and from each solution made up to 250 c.c. two quantities of 100 c.c. were taken, corresponding to 0.50 gm. of the original phosphate. Thus we get two series: the three solutions of Series *a* were precipitated with 10 c.c. of chloride of magnesium to estimate the phosphoric acid; and those of Series *b* with 10 c.c. of a solution of phosphate of ammonia at 10 per cent. to estimate the magnesia. At the same time, 50 c.c. of the original solution were submitted to direct precipitation and gave 0.2507 gm. of calcined precipitate (after standing sixteen hours). The following are the results obtained from the two series, *a* and *b*, after standing for sixteen hours.

	<i>a</i> .	<i>b</i> .	Difference: <i>b</i> - <i>a</i> .
I.	0.2508 gm.	0.2700 gm.	0.0192 gm.
II.	0.2506 "	0.2738 "	0.0232 "
III.	0.2498 "	0.2727 "	0.0229 "

These results were most unexpected. It certainly appears as if the Series *a* corresponds with the usual method for the estimation of phosphoric acid, since the concordance between the direct estimation and the result (I., *a*) which corresponds to it are as close as possible; but II., and above all III., in spite of the excess always caused by rapid precipitation, contain less phosphoric acid. This excess would appear, therefore, to be due to an excess of magnesia; and this would be confirmed by the figures of column *b*, which we may consider as proportional to the quantities of magnesia in the original precipitates.

The considerable differences between the corresponding figures of columns *a* and *b*, which should be identical, or at least very close to each other, present an important question to be solved.

1. Either in the original precipitates, and in Series *a*, there is a large excess of magnesia. It would then be necessary that, in the method as usually practised, tri-magnesian phosphate be formed; the estimation of the phosphoric acid being in such a case tainted by a serious error.

2. Or, in Series *b*, there is an excess of phosphoric acid. The weighed precipitate would then contain metaphosphate mixed with pyrophosphate, and to explain this fact it is necessary to admit that bi-ammoniacal magnesian phosphate is precipitated at the same time as the normal ammoniaco-magnesian phosphate. The estimation of the magnesia would then be erroneous.

3. Or, further, that the two causes of error co-exist. To decide which of these three hypotheses is correct, the preceding experiments are not sufficient, and it became necessary to have recourse to rigorous synthetical trials.

VII.—Trials with a Known Quantity of Phosphoric Acid.

It is not advisable to use ammonio-magnesian phosphate to obtain a known quantity of phosphoric acid; phosphate of ammonia is far preferable. But if we can make sure of the purity of this body, in so far that it contains nothing but phosphoric acid and ammonia, its hygrometric state and its degree of basicity need not be rigorously defined. So as to start from an absolutely certain base, after having made a solution containing about 34 grms. per litre of pure phosphate of ammonia, I caused 10 c.c. of this solution to be absorbed by pure magnesia, strongly calcined, and after drying at 100° it was again calcined (the calcination should last about three-quarters of an hour). The difference between the two weighings

gives, for 10 c.c., 0.1801 of phosphoric acid, with possible variations of one-tenth of a m.grm., more or less. Other trials made with 20 and 30 c.c. gave proportional results, with the same limits of error. We can therefore feel sure of the titration value of the solution.

Samples of 10 c.c. each were precipitated under the ordinary conditions by the addition first of 40 c.c. of water, then of definite quantities of citrate of ammonia and chloride of magnesium. The average of these experiments gave a weight of pyrophosphate of 0.2823 gm., with variations not exceeding two-tenths of 1 m.grm., more or less. This corresponds to 0.1802 gm. of phosphoric acid. From this we conclude that the precipitation is complete, and that the pyrophosphate of magnesia is of normal composition under the afore-mentioned conditions; that is to say, after sixteen hours standing in a volume of 150 c.c. containing 10 grms. of citric acid and at least one-third of its volume of free ammonia, and, finally, an excess of magnesia.

I have repeated the experiment under the same conditions, but using hydrochlorate of ammonia instead of citrate. This seems permissible, since there is no other base but magnesium to keep in solution, but the results cannot be depended on, and there is always a large excess. The weight of the calcined precipitate is increased to 0.2976 gm., which corresponds to 0.1903 gm. of phosphoric acid, taking it to have the composition of the pyrophosphate. This precipitate thus contains such a large proportion of tri-magnesian phosphate that even standing for sixteen hours does not suffice to transform it into ammoniaco-magnesian phosphate. This observation is of importance, inasmuch as it is often recommended to precipitate phosphoric acid after the molybdc separation, for example, by a simple addition of hydrochlorate of ammonia and of magnesia. Results obtained in this manner are quite untrustworthy.

VIII.—Experiments with a Known Quantity of Magnesia.

A known quantity of strongly calcined magnesia was dissolved in hydrochloric acid, then precipitated in the presence of citrate of ammonia by an excess of phosphoric acid; the quantity of phosphoric acid in excess was varied by using increasing quantities of a solution of 100 grms. per litre of phosphate of ammonia (titrating 0.54 gm. of phosphoric acid per 10 c.c.).

Two series of experiments were made:—

A on 0.0948 gm. of magnesia.
B " 0.1034 " "

In each series three precipitations were made, with 5, 10, and 20 c.c. of the solution of ammonic phosphate respectively. The following results were obtained:—

	<i>A</i> .	<i>B</i> .
Magnesia used	0.0948 gm.	0.1034 gm.
Corresponding pyrophosphate	0.2630 "	0.2868 "
Weight of calcined precipitate obtained with—		
5 c.c. ammonic phosphate	0.2687 "	0.2929 "
10 " "	0.2709 "	0.2981 "
20 " "	0.2779 "	0.3107 "

We can see from the above that even with a slight excess of phosphoric acid (5 c.c. leaving not more than 0.1 of phosphoric acid in the solution) there is an excess—due, without doubt, to the mechanical carrying down of the phosphoric acid by the precipitate. This excess further increases rapidly as the solution is richer in phosphoric acid.

For the purpose of confirming this, a third series, C, was made under the same conditions as B; but instead of weighing the precipitates at once, they were dissolved for the purpose of estimating the phosphoric acid in the ordinary manner. The following results were obtained:—

	C.
Magnesia used.. .. .	0.1034 grm.
Corresponding phosphoric acid	0.1834 "
Phosphoric acid obtained in the precipitate by—	
5 c.c. ammoniac phosphate	0.1940 "
10 " " "	0.1971 "
20 " " "	0.2064 "

It is seen thus that the precipitate carries down a large excess of phosphoric acid, which, on calcination, gives metaphosphate.

These results confirm and explain those which have been already obtained, notably in Section V.

Although I have not been able to isolate a bi-ammoniacal magnesian phosphate, everything points to the existence and formation of this compound, the more abundantly as the reaction takes place in a greater excess of ammoniac phosphate.

IX.—Conclusions.

Finally, we deduce from what has been stated the following consequences:—

1. The estimation of phosphoric acid, in the state of pyrophosphate, without any precautions beyond the preliminary elimination of the silica, gives trustworthy results, untainted by systematic errors.
2. Rapid precipitations cause an excess, due to the partial formation of trimagnesian phosphate, which is only transformed into ammonio-magnesian phosphate after sixteen hours contact with sufficiently concentrated citrate of ammonia (10 grms. of citric acid to 150 c.c. of solution). It therefore follows that to obtain satisfactory results the solution should be allowed to stand all night.
3. In spite of all this, the excess is sufficiently small as not to completely condemn these rapid methods, which can with advantage be used commercially if due notice is given that this is being done.
4. The transformation of the trimagnesian phosphate into ammonio-magnesian phosphate is very slow in the presence of hydrochlorate of ammonia only, and it should always be remembered to add the necessary quantity of citrate.
5. The precipitation of the magnesia in the presence of an excess of ammoniacal phosphate gives, at the same time, with the ammonio-magnesian phosphate another phosphate, not only poorer in magnesia, but also poorer as the excess of phosphoric acid present is greater. The estimation of the magnesia by this almost classic method is, therefore, always erroneous. This is a subject to which we shall return later on.—*Bull. Soc. Chim.*, vols. xviii., Nos. 16 and 17, 1897.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, November 4th, 1897.

(Continued from p. 263).

DISCUSSION.

DR. PERKIN said he felt much interested in the paper, because of the remarkable magnetic rotation of combined fluorine, for example, in fluorobenzene. When one atom of hydrogen in benzene is displaced by chlorine, the rotation is considerably increased. The substitution of bromine causes a still higher rotation, and that of iodine the highest. On the other hand, the substitution of fluorine reduces the magnetic rotation. He had suggested that this might be accounted for if fluorine were paramagnetic, because its magnetic rotation would then be the reverse of that of carbon and hydrogen. This, however, does not seem to be a probable explanation since it is now found that liquid fluorine is not paramagnetic. It

is possible that this element may have different values depending on whether it is free or combined. The nitro-group (NO_2) influences magnetic rotation much in the same way as fluorine.

DR. GLADSTONE remarked on the importance of Prof. Dewar's communication, the most interesting portion to him being that on the optical properties of the liquid fluorine. The specific refraction of that element had been calculated by him and his brother from fluorobenzene and from many salts, crystallised or in solution, with the invariable result that it was exceedingly small. In the last list of the specific refractions of the elements (*Proc. R. S.*, 1897, ix., 141) it is given at only 0.031, which is not a third of the next lowest in the list. Its specific dispersion is also low, and it has the additional peculiarity of giving a reversed spectrum. Now Prof. Dewar finds that liquid fluorine has about the same refractive index as that of amber; this is known to be 1.55 or thereabouts. As the specific gravity of the fluorine is stated to have been 1.14, we can easily calculate the specific refraction, viz., 0.482. This figure, instead of being the lowest in the list of elements, is nearly the highest, there being only six with higher values.

It is true that in some cases the specific refraction of an element in the free state differs somewhat from that deduced from its compounds. Fluorine would naturally be compared with the three halogens, chlorine, bromine, and iodine. Liquid chlorine has a specific refraction of about 0.27; in combination 0.28. Bromine has a specific refraction of about 0.20; in combination 0.21. Iodine vapour has a specific refraction of about 0.19; in combination 0.21. The free element, therefore, does not differ widely in specific refraction from the same element when in combination, and in each case is the smaller and not the greater of the two. A certain analogy does exist between fluorine and sulphur or phosphorus. These two when melted have high specific refractions, sulphur being 0.50 and phosphorus 0.59: these high figures are generally much reduced when the elements are in combination, but the extent of this reduction is by no means comparable with what would appear to be the case with fluorine. Although Professor Dewar's method is correct in principle, Dr. Gladstone expressed a strong hope that accurate determinations would be made by one or other of the more direct methods.

DR. THORPE said, in reference to the allusion by the President to his determination of the specific molecular volume of fluorine as far back as 1880, that too much stress could not be laid upon the particular value, viz., 9.2, which he then obtained. It was deduced from a study of the specific gravity and thermal expansion of arsenic fluoride, a substance which is not easy to obtain pure, and which is not altogether without action on glass, especially at temperatures approaching the boiling-point. It, moreover, presupposes that arsenic fluoride has a molecular constituent analogous to that of arsenic chloride. Such an assumption is probable; but having regard to the remarkable complexity of many fluorine compounds, as, for example, hydrogen fluoride itself, when compared with the corresponding chlorine compounds, the supposition cannot at present be regarded as more than probable. The particular value obtained, however, clearly indicated the order of the magnitude, as shown by its substantial agreement with the other values quoted by the author.

With respect to the question raised by Dr. Gladstone he might say that the peculiar behaviour of glass when immersed in arsenic fluoride was significant, and suggested a method by which the refractivity of liquid fluorine in the free state might be ascertained with a fair approximation to accuracy, viz., on the same principle as that adopted by the authors in determining the relative density of liquid fluorine—that is, by immersing solids of known refractivity in the liquid, and observing which became invisible. Arsenic fluoride is a highly refractive liquid, and some specimens of glass threads and tubes become almost invisible when immersed in it.

Professor DEWAR, in reply, observed that he did not intend to convey the impression that, because amber in liquid fluorine might be difficult to define clearly, it necessarily followed that the refractive index would turn out to reach 1.55, as Dr. Gladstone seemed to infer. His present impression was that it exceeded that of liquid air, but he could go no further. No doubt the next time Professor Moissan and he had the opportunity of continuing the experiment, a direct determination of the refractive index would be made.

*112. *The Liquefaction of Air and the Detection of Impurities.* By Professor DEWAR.

In a paper on "The Relative Behaviour of Chemically-prepared and of Atmospheric Nitrogen," read before the Society in the year 1894, it was stated that all samples of

holds the liquid air maintained under continuous exhaustion. As this low temperature had to be kept steady for from one to two hours, while at the same time the bulb B had to be completely covered with liquid air, it was necessary to arrange some means of keeping up the liquid air supply without disturbing the apparatus. The plan adopted is shown at H, which is a valve arrangement which can be so regulated as to suck liquid air from the large vacuum vessel A, and discharge it continuously along a pipe into the vacuum test-tube G, the latter being kept under good exhaustion. In working the apparatus the tube I is connected to a gasometer containing 10 cubic feet of air, so that the volume of air condensed in each experiment may be observed. This was generally from 2½ to 3 cubic feet. If there is a very small proportion of some substance not liquefiable or soluble in liquid air

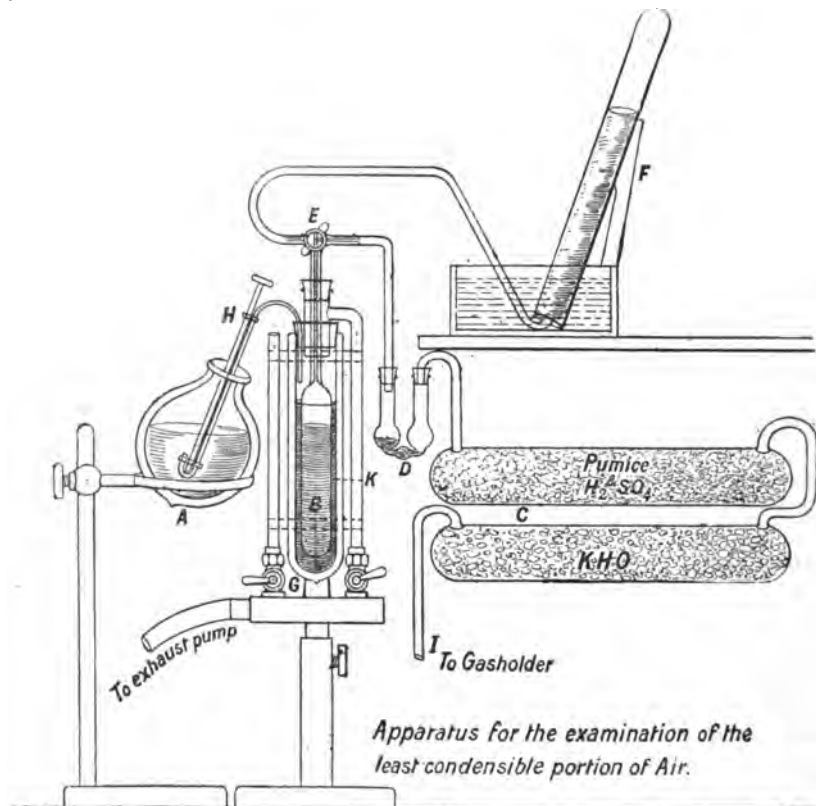


FIG. 2.

nitrogen and oxygen properly purified, are, when liquefied, clear transparent liquids, so that the solid matter which always separates when air or nitrogen or oxygen is liquefied on the large scale consists of impurities. Ordinary air, containing 4 parts of carbonic acid per 10,000 parts, gave a turbid liquid from the solidification of the carbonic acid; and oxygen containing traces of chlorine behaved in a similar manner. With the object of ascertaining the proportion of any gas in air that is not condensable at about -210°C . under atmospheric pressure, or is not soluble in liquid air under the same conditions, the following apparatus has been devised:—A cylindrical bulb of a capacity of 101 c.c., marked B in figure, had a capillary tube sealed into it terminating in a three-way stopcock, as shown at E. The parts marked C and D consist of soda-lime and sulphuric acid tubes for removing carbonic acid and water. The stand marked O holds the large vacuum test-tube into which B is inserted which

then we should expect the vessel B would not fill up completely into the capillary tube. This is, however, exactly what does take place. After forty minutes' cooling, the vessel B and the cool part of the tube were filled with liquid. In this experiment some 80 litres of air were condensed, and any accumulated uncondensed matter must have been concentrated in the upper part of the capillary tube which had a volume of 0.5 c.c. Under the conditions, therefore, the material looked for must be less than 1 part by volume in 180,000 of air.

To test the working with an uncondensable gas added to air, a volume of 10 cubic feet was taken in the gas-holder, and to that 500 c.c. of hydrogen were added. This is in the proportion of less than 1 in 500. Even after two hours' cooling, the tube B could only be filled four-fifths. In order to prove that the gas accumulated in the upper part of B was hydrogen, the three-way stopcock at E was turned, and the temperature allowed to rise

so that the gas was expelled from the evaporation of the liquid air and collected over mercury as shown at *F*. The gas thus collected was easily combustible, and consisted chiefly of hydrogen. The amount of hydrogen was then reduced to 1 part in 1000 of air, and it was found that after one and a quarter hour's cooling the bulb *B* had filled to within a $\frac{1}{2}$ c.c. of the capillary tube. A new sample of air containing 1 part of hydrogen in 10,000 of air filled the bulb *B* completely as if it were ordinary air.

It appears from these experiments that 1 part of hydrogen in 1000 of air is just detectable by this plan of working. As the 80 litres of air condensed contained some 80 c.c. of hydrogen, it appears that 100 c.c. of liquid air at from -200° to -210° C. had dissolved nearly all this gas; in fact, that 20 c.c. of hydrogen at the low temperature is dissolved in 100 c.c. of liquid air. In the

precipitate by transmitted light looked yellow-brown. This solid turns out to be of organic origin, probably of the petroleum order of compounds. It has a very marked aromatic smell resembling such bodies. The trace of material left gave, after treatment with concentrated nitric acid, the smell of nitrobenzene; and as its detection cannot be explained by the presence of any material of the kind in the vessels used in collecting, it must be assumed to be a normal constituent of the Bath gas. A further quantity of the Bath gas must be collected in order to confirm the presence of such bodies and to definitely make out their nature. Another peculiarity of the liquid is that, on examining it with the spectroscope, even through a thickness of 2 inches, no trace of the characteristic oxygen absorption spectrum could be detected. In all attempts to make nitrogen for liquefaction on the large

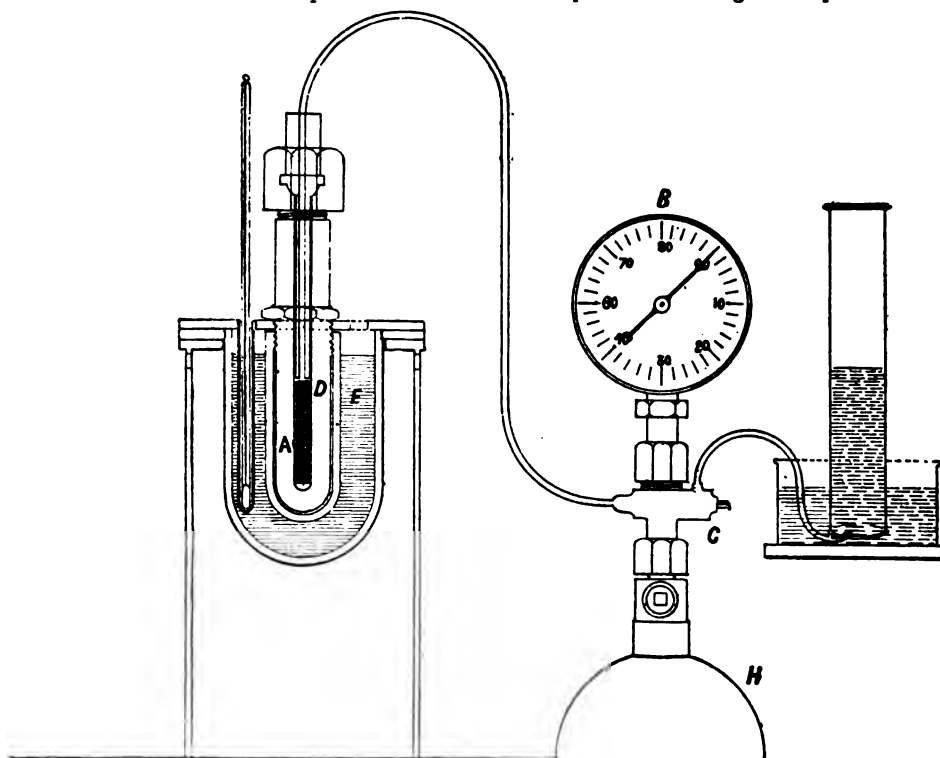


FIG. 3.

paper on "The Liquefaction of Air and Research at Low Temperatures" (*Proc.*, 1895, xi., 221) it was shown that if hydrogen containing a small percentage of oxygen were employed for the purpose of getting a hydrogen jet, the liquid collected from it was oxygen, containing, however, so much hydrogen dissolved in it that the gas coming off for a time was explosive.

In order to press this inquiry a little further, some natural gas known to contain a different constituent like helium suggested itself as being worthy of trial. Lord Rayleigh's results of the examination of the gas from the King's Well at Bath showed that it contained 1.2 part of helium per 1000 volumes, so that it seemed admirably adapted for such experiments. The author has to express his thanks to the Corporation of Bath for giving permission to collect samples of the gas.

The sample of gas from the Bath Spring was treated exactly in the same way as the hydrogen mixtures described above. During the liquefaction there was a marked difference in the appearance of the liquefied gas, for while the hydrogen and air mixtures gave a clear, transparent liquid, the product from the Bath gas was turbid, and the

scale, oxygen could always be detected in the liquid with the greatest ease by means of its absorption spectrum. After the cooling had continued for one hour the gas ceased to flow into the condensing vessel, and some 20 c.c. at the upper part of the glass cylinder *B* was filled with a gas that had not undergone liquefaction or solution. About 70 litres of the Bath gas were condensed, certainly the largest quantity of this gas ever subjected to chemical examination. This was boiled off just as the hydrogen was treated in the experiments described above, and as, by accident, too much nitrogen had volatilised along with the gas, oxygen was added and the mixture sparked over alkali to get rid of the excess of nitrogen. During the sparking the helium lines were well marked (along with others the origin of which must be settled later), and a vacuum tube filled with the product of the sparking gave a splendid spectrum of the gas. The sample of gas directly collected from the liquid nitrogen contained about 50 per cent of helium. It is therefore possible to separate helium from a gas when it is only present to the extent of one-thousandth part by liquefaction in the manner described. From this it would appear that helium is less

soluble in liquid nitrogen than hydrogen is in liquid air, and is of greater volatility than either of the constituents of air, as Professor Olzewski found (*Bull. Ac. Crac.*, 1896, 297) by direct experiment on a pure sample of the gas sent to Cracow by Professor Ramsay with the object of liquefaction. In the author's lecture (*Proc. Roy. Inst.*, 1896), entitled "New Researches on Liquid Air," the following observation occurs:—"The exceptionally small refractive value observed by Lord Rayleigh in the case of helium shows that the critical pressure of this body is proportionately high. It would therefore be more difficult to liquefy than a substance having about the same critical temperature but possessing a lower critical pressure than hydrogen." Now that it has been shown by Professor Moissan and the author that two substances like fluorine and argon, differing by two units in molecular weight, boil at nearly the same temperature, it seems reasonable to extend the analogy to the case of hydrogen and helium where the same difference occurs, and to suggest that they also probably have about the same volatility. If the sample of uncondensed gas resulting from the first liquefaction of the Bath gas were again treated in the same way, a much more concentrated specimen of helium could be obtained. Provided helium were wanted on a large scale, then a liquid air apparatus similar to that in use at the Royal Institution, transported to Bath and worked with the gas from the King's Well, could be made to yield a good supply. With a modified form of apparatus, it will be possible to collect any residuary gas from the use, not of 3 cubic feet of air or Bath gas, but from hundreds of cubic feet of such products. This investigation will be continued with new samples, in order to see if the composition of the gases changes and to isolate the hydrocarbons.

The author has to thank Mr. Lennox and Mr. Heath for able assistance in carrying out the experiments.

DISCUSSION.

SIR WILLIAM CROOKES said that a few days ago he received from Professor Dewar a tube containing some of the gas at atmospheric pressure. A small quantity was let into a new and completely exhausted spectrum tube, which was then re-exhausted and filled several times. On exhausting to 5 m.m. pressure and passing an induction spark it showed the nitrogen spectrum brilliantly, and on intercalating a condenser the yellow helium line was visible, but too faint to be measurable in the large spectro-scope. To remove the nitrogen, 47 c.c. were mixed in an eudiometer with an equal volume of oxygen, and sparked for about eight hours, absorption of the products being effected by strong potash solution over the mercury. When contraction had ceased, the residual oxygen was absorbed by passing pyrogallol into the potash. The unabsorbed gas amounted to 25 c.c. This gas, dried over phosphoric anhydride, was examined in a new spectrum tube, end on. (Tube shown in action). It gave the helium line (wave-length 5875.87) brilliantly, together with the other helium lines. No argon lines could be seen.

*113. "The Absorption of Hydrogen by Palladium at High Temperatures and Pressures." By Prof. DEWAR.

One of the author's earliest papers was entitled "The Motion of a Palladium Plate during the Formation of Graham's Hydrogenium." The explanation of the motion, together with a record of other experiments, can be found in the *Proc. Roy. Soc. Edin.*, 1868, vi., 504.

A subsequent investigation by the author into the physical constants of hydrogenium appeared in the *Trans. Roy. Soc. Edin.*, 1876, xxvii., 167, and had reference to the specific gravity, specific heat, and coefficient of expansion of the occluded hydrogen. These observations led to the conclusion that the specific gravity was independent of the amount of condensed gas, and had a mean value of 0.62. The specific heat, relatively to palladium of the condensed hydrogen appeared to vary inversely as the quantity occluded, but taken relatively to successive

charges was nearly constant, having the value 3.4, which is identical with that of gaseous hydrogen at constant pressure. The coefficient of cubical expansion of the alloy is about twice that of palladium, and that of the hydrogen in its compressed state not more than three times that of mercury. A later communication was made to the Philosophical Society of Cambridge (*Proc.*, 1878, iii., 207) dealing with the thermo-electric relations and electric conductivity of hydrogenium. It was shown that the potential difference of a junction of hydrogenium-palladium is at ordinary temperature nearly equal to that of an iron-copper junction, and that it increases with the temperature according to the general parabolic law; the rate of the increase being, however, greater than iron-copper, and subject to a regular variation on account of successive heatings. The formation of thermo-electric piles, and of neutral points in a wire of this substance, along with the continuous formation of thermo-electric currents through the application of a hydrogen flame were explained. Experiments on electric resistance proved that it increases directly with the amount of hydrogen condensed in the palladium.

Subsequent investigators have dealt more elaborately with the many problems suggested by hydrogenised palladium, but so far the essential facts referred to above have been confirmed.

In the course of the early observations the following experiment is recorded as illustrating the absorption of hydrogen by palladium at a red heat:—

Take a strip of thin sheet palladium, 4 or 5 c.m. long and about 5 m.m. in breadth, clamp it firmly by the end in a suitable support, so that the strip is free to vibrate, and insert it edgewise in the middle of a hydrogen flame, burning from a nozzle about 1 m.m. in diameter. If the palladium be now depressed into the inner dark cone it immediately begins to vibrate, producing a low, musical note.

If the flame be extinguished by stopping the current of hydrogen for an instant, on allowing the gas to flow the vibration commences again, and may be kept up without any actual flame.

The motion in this position in the flame is due to the absorption of hydrogen on the cool side next the inner cone, with its attendant increase of length, producing a bending of the sheet into the hot portion of the flame, where the hydrogen is instantly expelled from the palladium, which is forced to return to its original position from its natural elasticity.

It is now known that no absorption of hydrogen at atmospheric pressure by palladium takes place above 145° C., so that the cause of motion must originate at a comparatively low temperature. The question arises,—Can palladium, under any condition of pressure, absorb hydrogen at a red heat in quantity at all comparable to what it can do at lower temperatures? If free hydrogen and palladium-hydrogen are compared as regards volatility, the one boils at 30° (abs.), the other at 420° (abs.) very much like two isomeric forms of the same substance. This ratio of 1 : 14 given (and certainly the ratio could not be made greater than 1 : 16, since the absolute boiling-points may be taken as in the ratio of their respective critical points), we thus arrive at a hypothetical palladium-hydrogen critical point of 640° (abs.) or 360° C. An almost exact parallel may be drawn between palladium-hydrogen in its relation to free hydrogen, and iridium oxide in its relation to free oxygen. Thus liquid oxygen boils at 90° (abs.) and the tension of dissociation of iridium oxide is 1 atmosphere at 1423° (abs.). The ratio of the absolute boiling-points of liquid oxygen and the oxygen of iridium oxide are therefore as 1 : 15.9, which is almost the same value as that found above for the relative volatilities of hydrogen and palladium. In either case, the ratio of the absolute boiling-points of the respective substances may be taken as approximately representing the ratio of the latent heats of transition of state. It might then be possible that palladium no longer absorbed

hydrogen under any condition of pressure. The present experiments were undertaken with the view of answering this question.

The diagram (3) shows the general arrangement of the apparatus most suitable for examining the behaviour of the metals like palladium, sodium, potassium, &c., towards hydrogen at high temperatures and pressures.

A rod of palladium, A, weighing about 119 grms., kindly placed at my disposal by Mr. George Matthey, F.R.S., was placed in a strong steel cylinder, D, having an accurately-fitting conical joint. As little extra space as possible was left in the cylinder, which was heated in a bath of fusible metal, E. The vessel was connected with the manometer, B, by a strong copper tube, and the latter was similarly joined to a compressed gas cylinder, H, containing hydrogen. The apparatus, without the palladium, must be carefully tested at high pressures and temperatures. There must be no trace of a leak. An extra stopcock at C enabled the hydrogen accumulated in the apparatus to be blown off suddenly when required, after the hydrogen cylinder stopcock was shut off. Before commencing the experiments at high temperatures, it is well to charge the apparatus to a pressure of 20 atmospheres with hydrogen, and then blow off the gas and measure it. In this way the volume of hydrogen that is absorbed for every diminution of the pressure of hydrogen is known. In the first experiments a pressure of 20 atmospheres of hydrogen in the apparatus corresponded to 780 c.c. of gas, measured at atmospheric pressure. When the fusible metal bath was heated to 420°, and hydrogen at a pressure of 80 atmospheres introduced at starting, it fell to a pressure of 60 atmospheres in two and a half minutes. Blowing off the gas instantly to get rid of accumulated impurities, and again applying a pressure of 80 atmospheres of hydrogen, the pressure was reduced to 60 atmospheres in six minutes. When the same operations were repeated a third time, the diminution of pressure by 20 atmospheres took sixteen minutes, and a fourth operation required 28 minutes. In all, therefore, upwards of 3000 c.c. of hydrogen were absorbed in less than an hour. If the palladium could be seen at a low red heat, then during the rapid absorption of the hydrogen as described in the last experiment, the temperature must rise very considerably, and the metal, during the operation, must actually appear to glow much brighter. Calculating from the tensions of the gas, the evolution of heat at 300° must be about 4698 grm.-units of heat per grm. of hydrogen absorbed. The reverse action would take place on reducing the pressure of hydrogen in the charged palladium. After the four charges the pressure remained constant at 80 atmospheres, no more hydrogen being absorbed. The hydrogen gas outside the palladium was now suddenly blown off, the stopcock shut, and the pressure allowed to rise from the escape of gas absorbed by the palladium. In this way, it was noted that a pressure of 40 atmospheres was reached in half an hour. The whole amount of gas that had been absorbed by the metal was found, on measurement, to be 2980 c.c. After the first charge of hydrogen, the steel cylinder was opened and the palladium examined. It was found to have a deep rent in it extending along nearly the whole length of the rod. During the occlusion of the hydrogen the volume of the metal is increased by one-tenth, so that in the passage of hydrogen in and out of the metal enormous strains must be produced. As the volume of the original metal is a little less than 10 c.c., it may be taken that above 300 times its volume of hydrogen had been absorbed at the temperature of 420° and under a pressure of 80 atmospheres. The free space in the manometer and connections was now diminished, so that a pressure of 20 atmospheres corresponded to a volume of 300 c.c. of hydrogen instead of 780 c.c. as above. The palladium was saturated at 360° C. under a pressure of 80 atmospheres in the manner described above, except that a very much larger number of charges of hydrogen had to be employed. After saturation, the pressure of

hydrogen was slowly reduced to 25 atmospheres: it rose to 30 atmospheres from gas passing outwards from the metal, now heated up to 500° C., and finally reached 100 atmospheres; on cooling to 400° the pressure diminished from re-absorption of the hydrogen. On blowing off the gas between 400° C. and 500° C., 1400 c.c. of free and 3300 c.c. of combined hydrogen were found. A rod of palladium, in this way, can be quickly charged with hydrogen at about 300° C. or 400° C., and as it is only the pure gas that is occluded, this process may be used as a rapid means of getting pure hydrogen in quantity for experimental purposes.

In the next experiment, the palladium was heated to 500° C. before any hydrogen under pressure was applied. No absorption was observed till the pressure of hydrogen reached 60 atmospheres. On charging as before at pressures between 80 atmospheres and 60 atmospheres, the metal was found to absorb 1900 c.c. of gas. The experiment was repeated, with the difference that the charging pressure of hydrogen was raised to between 120 atmospheres and 100 atmospheres, and it was found that the palladium had now occluded 3700 c.c. of hydrogen. Thus it appears from these experiments that at 500° C. palladium can still occlude 300 times its volume of hydrogen under a pressure of 120 atmospheres. The observations on the tension of hydrogen in palladium by Troost and Hautefeuille showed that, for the same temperature, the values became constant and independent of the amount of occluded gas, only when the volume of hydrogen absorbed lay between 200 and 600 times than of the metal. Any other proportions gave variable tensions for the same temperature. The fact that 300 volumes can still be occluded at 500° C. seems to show that palladium and hydrogen, under such conditions, still follow the same laws of absorption as at lower temperatures. Nothing analogous to a critical point, where no combination takes place between the metal and hydrogen, has been reached.

Hoitsema published an important paper on palladium-hydrogen tensions in the *Archives Néerlandaises*, 1896, xxx., 44. In this memoir, Hoitsema gives also a series of observations on the same subject made by Roozeboom. Taking the tensions given by the latter (simply because the curve seems more regular) for the horizontal portions of the dissociation curves at different temperatures, and calculating a Willard Gibbs' formula, from the following data, viz., 20° C. pressure 7 m.m.; 100° C. pressure 205 m.m.; 170° C. pressure 1467 m.m., the expression results (where T is the absolute temperature)—

$$\log. p = 7.00338 - \frac{1983.4}{T} + 0.2378 \log. T.$$

From this it follows that the latent heat of dissociation of the palladium-hydrogen per atom of hydrogen in grm.-units is $4561 + 0.2378 T$. This would seem to show the latent heat of dissociation increases instead of diminishing with temperature. In other words, the heat of combination should be rather greater at higher temperatures, instead of diminishing as it must do if a point where no occlusion takes place were being approached. Thus theory and experiment would seem to agree.

The best and safest method for the experimental study of the relations of hydrogen and palladium at high temperatures and pressures would be to investigate the change of electrical resistance in a heated wire of the metal when subjected to different hydrogen pressures. The problem is, no doubt, more complicated, still interesting results must follow from such an investigation. Some of the electrical properties of hydrogen and palladium at low temperatures have been determined by Professor Fleming and the author, and the results will appear in future publications bearing on the subject.

The author is indebted to Mr. Robert Lennox for able assistance in the conduct of the experiments.

DISCUSSION.

Mr. R. J. FRISWELL asked whether the President had made any measurements of the tensile strength of the

steel. He was astonished to hear of the metal standing 100 atmospheres at over 500° C. He was asking for information, as he had been unable to obtain any data as to the strength of metals near a red heat, a point at which it must be rapidly falling away. The matter was of great interest for experimenters using autoclaves. Engineers did not seem to have done any work on tensile strength at points above the temperatures usual in steam boilers.

Prof. DEWAR, in reply to Mr. Friswell, agreed that no engineering formulæ existed. The experiments were dangerous, but one had to take the risk. The metal used was Whitworth compressed steel, and the vessel was made by drilling out a solid mass. He had no data as to tensile strength, the results were desired and the risk taken.

(To be continued).

PHYSICAL SOCIETY.

Ordinary Meeting, November 26th, 1897.

Mr. SHELFORD BIDWELL, President, in the Chair.

MR. ROLLO APPLEYARD read a paper on "*The Failure of German-silver and Platinoid Wires.*"

The mechanical defectiveness, and the consequent electrical instability of alloys used for electrical wires, may be discussed from two points of view:—(1). As to the constitution and metallurgy of the alloy. (2). With regard to the subsequent treatment and environment of the wire. In stating the case, the author gives instances of the failure of German-silver and platinoid wire, that have occurred among several thousands of resistance-coils distributed over widely different latitudes. In periods of time, varying from six weeks to several years after manufacture, the wire on some of the bobbins became brittle, and broke, not only on the outer layers, but also within the coils. The towns where the faults appeared are all within the tropics, and included nearly within the isotherm of 25° C. Other coils, of nominally the same material, manufacture, and environment, have retained their original good condition. It follows that metallurgical differences exist between different samples of the same nominal quality of alloy. Examples are given to prove that failure sometimes occurs with platinoid through which no electricity has passed. Provided that the wire is good, the effect of environment is almost insignificant, i.e., the question is one of metallurgy rather than of instrument-making.

The author introduces a distinction in regard to brittleness. He discriminates between "primary" and "secondary" brittleness. "Primary" brittleness is characteristic of certain alloys (for instance of gold-lead or of gold-bismuth) from the moment of their solidification. But the brittleness of German-silver and of platinoid is of a different order; it is a subsequent phenomenon. "Primary" brittleness is thus an accident of birth, and "secondary" brittleness is a disease that develops with age and circumstance. The fracture of bad specimens of German-silver and platinoid shows patches of dark metal, crevices, and fissures. It may be supposed that, during the process of cooling, "liquation" occurs—the metals that first solidify rejecting yet molten portions, as ice rejects foreign matter. Consequently the strength of the final alloy varies from point to point of its mass, and in passing afterwards through the die the weaker portions give way, and the general structure is loosened. Moisture can then intrude through the capillary channels. At all fissures and crevices the electric current produces undue heating: this accounts for the failure of resistance-coils on arc-light and other circuits. As regards the protection of coils against moisture, paraffin-wax is of no use whatever; it is highly absorbent. Shellac varnish is greatly to be preferred. Ebonite does not seem to have

any deteriorating effect, but it may be well to keep the alloys out of actual contact with it.

In conclusion, Mr. Appleyard expressed a hope that British metallurgists would give electrical alloys special consideration. Already British cable-manufacturers are importing thousands of tons, annually, of sheathing-wire from Germany: this is sufficiently to be regretted; he had good reason to know that instrument-makers were beginning to get the wire for their resistance-coils also from Germany. He had not enough experience of manganin to say whether that material would stand rough service in the tropics.

Prof. AYRTON said the paper had raised the extremely interesting question of the permanence of metals used for resistance-coils. Some time ago he had immersed bare platinoid wires in running water in metal tanks, and the wires all broke in short pieces. He thought, at the time, this might be due to electrolysis. On another occasion he had found that, by raising the temperature of platinoid to a dull-red heat in the air, by an electric current, any acquired faults in the wire were corrected, and the original resistance and flexibility were restored. Even when such metals are in good condition, the resistance-temperature curve does not return upon itself; it encloses a loop, indicating two distinct values for resistance at each temperature. He had been told by Dr. Muirhead that coils intended for hot climates should be enclosed in air-tight metal-cases. English manufacturers were still dubious in regard to manganin. In 1892 he had twenty coils of this material, each of 1000 ohms; the wire was silk-covered. There were 2000 volts between the terminals. Their resistance had certainly not changed by 1 in 1000, although there was some amount of vagueness regarding the fifth figure, which might be due to molecular alteration, for they were heated more than was good for resistance-coils. He confessed that this manganin had come from Germany.

Dr. S. P. THOMPSON mentioned an alloy that was proposed in Germany under the name of "Constantine." He would like to know whether any information could be obtained as to the employment of cast-iron wire. It was a metal that in some respects commended itself. He had observed the failure of some German-silver coils, but he had generally attributed it to rough handling.

Mr. W. WATSON referred to the recent work done at the Reichsanstalt with regard to German-silver and platinoid. It was there found that all alloys containing zinc were liable to erratic change of resistance, and were unsuitable for standard coils. Moreover, even the slight amount of zinc introduced into manganin during soldering with soft solder robbed that alloy of its constancy. Silver solder, containing 75 per cent of silver, should be employed for manganin. If Prof. Ayrton's coils were soldered with soft solder, that was sufficient to account for the change in the fifth figure. Shellac varnish was undoubtedly the best protection for coils. Absolute alcohol should be used as the solvent, and the coils should afterwards be heated for some hours at 140° C. If a heating-current was passed through German-silver or platinoid coils immersed in water, the general result was to produce brittleness. Mr. Watson then described a thermostat which he had contrived for drying the coils after applying the shellac varnish. A hot-air oven contains a thermometer with a platinum contact at the 140° mark and an 8 c.-p. lamp. The thermometer is in circuit with a relay actuating a mercury-key for the 8 c.-p. lamp. The key consists of two mercury-cups, and a corresponding U-piece of copper, inverted, one limb to each cup. It is important to keep the heating-circuit always made; for this purpose a 32 c.-p. lamp is permanently connected between the two cups.

Prof. PERRY then read a note on a question in "*Thermodynamics*," arising from correspondence that had taken place between himself, Prof. Ramsay, and Mr. Rose-Innes, with regard to a paper in the *Roy. Soc. Trans.*

Mr. ROSE-INNES replied.

The PRESIDENT proposed a vote of thanks to the authors, and the meeting adjourned until Dec. 10th.

CORRESPONDENCE.

MOUNTING MICROSCOPIC OBJECTS.

To the Editor of the Chemical News.

SIR,—In the course of preparing the catalogue of the collection of slides belonging to the Royal Microscopical Society, I met with about a hundred anatomical preparations mounted in fluid, some in cells of considerable size. They are the work of the late R. J. Farrants, formerly a President of the Society, and were presented with his entire collection soon after his death. The date of the preparations in question is certainly previous to 1860.

I found the cells leaky, and the marine glue generally in bad condition; indeed, this was the weak point of the mounts; the cementing of the cover glasses was in nearly every case perfectly sound—as might be expected from such a master of technical details as the late Mr. Farrants.

It is certain that marine glue—so much in use many years ago for attaching cells to glass—is, in common with many other compounds of indiarubber, a somewhat unstable substance, and has evidently broken down after a period of from thirty to forty years.

The mounting fluids employed were dilute alcohol and sometimes a weak glycerin preparation, and both occasionally with some kreosote.

I have also found marine glue has failed in my own collection, and I now attach cells to glass with a mixture of red and white lead; artist's flake white, from the tube, worked up with the red lead until it is too stiff to use, and then mixed a little at a time with japanner's gold size. No more should be mixed at once than is sufficient to fix two or three cells, as the mixture rapidly becomes unworkable after the introduction of the gold size.

When the cells are in place, they should be put in a warm situation—I use the top of the hot-water cistern—and after a day or two the surplus cement should be scraped off; they are then left for a fortnight or three weeks to thoroughly harden; the final cleaning is done with a little alcohol. The cells are now impervious to ordinary mounting media.

When a cell contains an appreciable quantity of fluid, I adopt the unprofessional plan of leaving a small air bubble, which acts as a spring and saves the cell from rupture at some weak point, which sooner or later happens when cells are entirely filled with fluid.—I am, &c.,

W. T. SUFFOLK, Treas. R.M.S.

143, Benlah Hill, Norwood, S.E.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xvii.-xviii., Nos. 16-17.

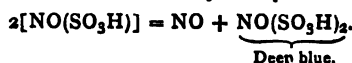
Observations on the Spectra of Compound Bodies.—A. de Gramont.—The author having noticed that many mistakes may be made by using salts, oxides, or solutions haphazard, has found that the spectra given by such compounds can be divided into two fundamental classes:—(1), the line spectra, and (2) the band spectra. The first he calls *atomic*, the second, owing to the lower temperature at which they are seen, he calls *molecular spectra*.

Amongst other interesting facts M. de Gramont finds that the proportion of an element in a compound can be recognised in three distinct manners:—1st, by a persistent spectrum, reduced to certain principal lines; 2nd, by a fleeting spectrum of unequal duration for any particular element; 3rd, by an intermittent and irregular spectrum. It should be borne in mind that the sensibility of the spectral reactions is entirely variable from one element to another.

Dissociation Spectra of Melted Salts. Alkaline Metals: Sodium, Lithium.—A. de Gramont.—Already inserted in full.

Dissociation Spectra of Melted Salts. Alkaline Metals: Potassium.—A. de Gramont.—Already inserted in full.

On Blue Nitrosodisulphonic Acid and some of its Compounds.—P. Sabatier.—In the reaction between oxide of copper and a solution of nitrate of silver the author has observed that the product, when treated with concentrated sulphuric acid, gives a very intense purple-blue colouration, which disappears on the addition of water. He finds that the principal cause of the colouration is due to the acid existing in the solution, this being due also to the presence either of nitrogen or of sulphur. In attempting the synthesis of nitrosodisulphonic acid the author tried several methods before arriving at one which now appears to be the most practicable. He effects the reaction by means of a carefully diluted sulphuric acid, previously saturated with sulphurous acid, and kept at 0°. The mixture of nitric oxide and air, when first added, gives no colouration, but after a time, varying within certain limits, there occurs a lively ebullition of the liquid, accompanied by a violent colouration, so intense as to be almost opaque. The final reaction may be expressed as—



Among the properties of nitrosodisulphuric acid may be noticed that it is quickly decolourised by agitation with air, peroxide of hydrogen, or persulphuric acid; chlorine gives a similar result, as does bromine, but more slowly; iodine has apparently no effect; the alkaline chlorides are violently decomposed, with disengagement of hydrochloric acid gas and free chlorine. Sulphurous acid direct has no apparent action on the nitrosulphuric solution, even when concentrated, but when diluted by one-fifth of its volume of water the blue compound is immediately obtained. The research tends towards the preparation of the various salts of nitrosodisulphuric acid apart from the preliminary solution in concentrated sulphuric acid.

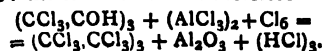
Dissociation of Minium.—H. Le Chatelier.—Experiments made with minium, free from carbonate of lime and other impurities, capable of giving off gas, show that there is still a gas evolved when heated up to temperatures varying from 445° to 636°; from which it appears that in ordinary air carrying oxygen at a tension of 150 m.m., minium can be formed at a temperature below 550°, but the temperature of most rapid oxidation is below but very close to 500°.

Impurities of Commercial Carbides of Calcium.—H. Le Chatelier.—Already inserted in full.

Action of Chlorine on Chloral in the presence of Chloride of Aluminium.—A. Mouneyrat.—The formation of the hexachlorethane is due to the fact that, by the action of chloral and chloride of aluminium, there is first of all formed a pentachlorethane, according to the equation—



this, under the influence of chlorine in the presence of chloride of aluminium, gives hydrochloric acid and hexachlorethane: the total reaction is therefore—



Action of Bromine on Chloral in the presence of Chloride of Aluminium.—A. Mouneyrat.—The author first thought that by acting on chloral in the presence of chloride of aluminium with bromine he would obtain a mixed chlorobromided compound; but he found that the body obtained did not contain any bromine in its molecule. He also tried the action of iodine under the same circumstances, but with similar results; he has obtained nothing but the hexachlorethane, C_2Cl_6 .

MISCELLANEOUS.

The Royal Society.—At the Anniversary Meeting on November 30th, the following Council and Officers were elected for the ensuing year:—

President—Lord Lister, F.R.C.S., D.C.L.

Treasurer—Sir John Evans, K.C.B., D.C.L., LL.D.

Secretaries—Prof. Michael Foster, M.A., M.D., D.C.L., LL.D.; Prof. Arthur William Rücker, M.A., D.Sc.

Foreign Secretary—Sir Edward Frankland, K.C.B., D.C.L., LL.D.

Other Members of the Council—Prof. William Grylls Adams, M.A.; Prof. Thomas Clifford Allbutt, M.D.; Sir Robert Stawell Ball, M.A.; Rev. Thomas George Bonney, D.Sc.; Prof. John Cleland, M.D.; Prof. Robert Bellamy Clifton, M.A.; Prof. James Alfred Ewing, M.A.; Alfred Bray Kempe, M.A.; John Newport Langley, D.Sc.; Joseph Larmor, D.Sc.; Prof. Nevil Story Maskelyne, M.A.; Prof. Raphael Meldola, F.C.S.; Prof. Edward Bagnall Poulton, M.A.; William James Russell, Ph.D.; Dukinfield Henry Scott, M.A.; Prof. Walter Frank Raphael Weldon, M.A.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Wheat Phosphates.—Will some correspondent kindly give me address of a manufacturer of wheat phosphates, or the name of a book or pamphlet describing the process of producing them from bran.—CATO.

MEETINGS FOR THE WEEK.

MONDAY, 6th.—Royal Institution, 5. General Monthly Meeting.
— Society of Chemical Industry, 8. "The Sulmau-Teed Process of Gold Extraction," by H. L. Sulman and Dr. F. L. Teed.
— Society of Arts, 8. (Cantor Lectures). "Gutta Percha," by Eugene F. A. Obach, Ph.D., F.C.S.
WEDNESDAY, 8th.—Society of Arts, 8. "The Mining and Metallurgical Industries of Sweden as shown at the Stockholm Exhibition of 1897," by Bennett H. Brough.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1985.

ON THE ELECTROLYTIC SEPARATION OF NICKEL AND COBALT FROM IRON. ITS APPLICATION TO THE ESTIMATION OF NICKEL IN STEELS.

By O. DUCRU.

I. THE complete separation of nickel and cobalt from large quantities of iron presents considerable difficulties, and the large number of methods already published does not yet show that there is any satisfactory method known for effecting it.

The principal difficulty arises from the fact that the ferric compound (hydrate, basic acetate, &c.) always contains a considerable proportion of other metals (Ni, Co, Mn, Cu, &c.): to obtain a complete separation we are therefore obliged to multiply the precipitations, which not only takes up a great deal of time, but also leads to the accumulation of inconveniently large volumes of liquid.

II. For some years past, owing to their special properties, nickel steels have taken an important place industrially. A rapid and accurate method for the determination of nickel in these alloys is therefore of some interest. In his masterly work on the "Methodes d'Analyse des Fers, des Fontes, et des Aciers," carried out by the desire of the Commission on the Testing of Materials for Construction, M. A. Carnot, for this purpose, gave preference to the method proposed by Rothe. This method, also suggested in France by M. Hanriot, rests, as is well known, on the separation of ferric chloride from its acid solution, by means of ether. Latterly M. Pinuera has modified this method of separation, and uses ether saturated with hydrochloric acid at a low temperature (E. Pinuera, *Comptes Rendus*, 1897, vol. cxxiv., p. 124).

III. It is easy to achieve the same result by means of electrolysis, by paying attention to the following remarks; if we precipitate a ferric solution containing, for example, nickel, with an excess of ammonia, a part of the latter metal will remain in solution, while a considerable amount is carried down with the ferric hydrate.*

If, however, we submit the ammoniacal liquid holding the precipitate in suspension to the action of electrolysis, we obtain the integral deposition of nickel on the cathode. The separation is not absolutely complete, as a small quantity of iron is also nearly always deposited on the cathode; but under suitable conditions this quantity is nearly constant; it varies from 1 to 2 m.grms., while the iron present may be about 400 or 500 m.grms. For exact work, then, it is necessary to make a correction in the weight of metal deposited: this is easily done by dissolving in hydrochloric acid, and, after peroxidation, precipitating by ammonia.

IV. The use of a nitric solution, which under analogous conditions enabled M. Riche (*Ann. Chim. Phys.*, series 5, vol. xiii., p. 528, 1878) to separate copper from iron, presents certain difficulties. It is the same with hydrochloric solutions. Good results can be obtained by using a sulphuric solution, containing sulphate of ammonia. The following is the method of procedure:—

To the solution containing the nickel and a maximum of iron is added a slight excess of sulphuric acid; it is then evaporated to dryness; the residue is taken up with

the smallest quantity of water possible, 5 to 10 grms. of sulphate of ammonia is added, and the solution warmed until it becomes clear. This solution, while agitating, is poured into the crucible of a Riche apparatus, in which 60 or 70 c.c. of concentrated ammonia has already been placed. We then proceed with the electrolysis. Two or three accumulators, coupled in series so as to obtain from 1.5 to 2.5 ampères at the commencement, form a convenient source of electricity.* Under these conditions, after about four hours, the nickel is entirely deposited.

V. This method has been checked by means of titrated solutions of iron and nickel, and some of the results obtained were as follows:—

No.	Fe m.grms.	Ni added m.grms.	Metal deposited m.grms.	Correction (iron) m.grms.	Ni recovered m.grms.	Difference m.grms.
1.	404.2	29.8	30.8	1.0	29.8	0.0
2.	269.5	74.6	75.9	1.3	74.6	0.0
3.	269.5	149.2	150.1	0.8	149.3	+0.1

The same method may be applied equally well to cobalt:—

No.	Fe m.grms.	Co added m.grms.	Metal deposited m.grms.	Correction (iron) m.grms.	Co recovered m.grms.	Difference m.grms.
4.	404.2	62.5	63.4	1.9	61.5	-1.0

VI. Estimation of Nickel in Steels.—250 to 300 m.grms. is dissolved in *aqua regia* in a porcelain crucible. When this is completed we add 1 c.c. of sulphuric acid, and evaporate until white fumes begin to be evolved. We then proceed as above. The following results were obtained in the analysis of a series of steels, containing from 1 per cent to 50 per cent of nickel:—

No.	Approximate quantity of present sample per cent.	Weight of sample.	Metal deposited m.grms.	Percentage found.	Correction.	Corrected percentage found.	True percentage present.
5	1	256.5	3.1	1.21	0.35	1.07	1.00
6	10	434.0	44.1	10.16	0.70	10.00	10.00
7	20	297.5	59.7	20.10	1.10	19.75	20.00
8	25	295.9	76.6	25.95	0.50	25.75	25.55
9	50	230.7	114.8	49.80	0.35	49.40	49.57

The figures given in the column *True percentage present* were obtained by a series of precipitations by ammonia in considerable excess, until the solutions no longer gave the nickel reaction with sulpho-carbonate of potash; 1 gm. of metal was used, but four or five precipitations were necessary to obtain a complete separation. The ammoniacal solutions were mixed, evaporated, and precipitated while boiling by caustic soda; the nickel was then estimated electrolytically by the method of Fresenius and Bergmann.

An examination of this table shows that in actual practice it would often be unnecessary to make the correction, a sufficient approximation being found by considering the total weight of metal deposited to be nickel; the results thus obtained, in the column headed *Percentage found* are within 0.5 per cent of being correct.† The estimation of nickel in a steel of any percentage can be effected in a few hours.

VII. Experience shows that it is unnecessary to separate the silicon or carbon; neither the small quantities of manganese and phosphorus (the sample of steel marked No. 6 contained 0.52 per cent Mn, and traces only of P) found in steels, nor the presence of chromium (the sample marked No. 7 contained 28 per cent Cr.) interfere with the use of this method; but traces of manganese are nearly always found with the small quantity of iron on the

* This preparation reaches 27 per cent for nickel, and 48 per cent for cobalt, in the experiments described by Baumhauer (*Archives Néerlandaises*, 1890, vol. vi.), and under some conditions it may be considerably higher.

* That is to say, 25 to 45 milliampères per square centimetre for the acting part of the cathode, assuming that the density of the current is uniform.

† Cobalt, if present, is reckoned as nickel according to the trade custom; the small quantity of copper (1 to 2 milligrammes) which is generally present in nickel steels is without influence.

cathode. To obtain an exact correction we precipitate the two metals at once, by adding, as recommended by M. Carnot, a little peroxide of hydrogen to the solution of the deposited metals, supersaturating with ammonia, and boiling. As the coefficients of the transformation—

$$\frac{\text{Fe}}{\text{Fe}_2\text{O}_3} = 0.700 \text{ and } \frac{\text{Mn}}{\text{Mn}_2\text{O}_4} = 0.721$$

are very close, there is not room for much error in applying either of them to weights of oxides of about 2 m.grms. It is in this manner that the figures in the column marked *Correction* were obtained.

VIII. It is only natural to suppose that the small quantities of iron present on the cathode are due to the reduction, by contact with it, of the ferric and ferrous hydrates soluble in ammonia. However, no deposition is obtained by the electrolysis, under the conditions mentioned, of the ferric hydrate alone. Further, electrolysis carried on for four, or even sixteen hours, have given the same results.

On the other hand, if we add to the ferric solution notable quantities of manganese or of phosphoric acid, the proportion of iron deposited becomes considerably augmented, and the method can no longer be trusted; the traces of manganese, almost always contained in pure solutions, must therefore be carefully dealt with; as manganese easily changes from its state of oxidation, when in the near neighbourhood of the electrodes, it might without difficulty reduce some of the ferric hydrate to the ferrous state. To decide this question a ferric solution was prepared by decomposing ferrocyanide of potassium, purified by crystallisation, by sulphuric acid, and precipitating the iron in the state of sulphide: this solution was perfectly free from both manganese and phosphoric acid. It was with this that the above-mentioned experiments, 1, 2, 3, and 4, were performed, all of which gave a small quantity of iron. It is therefore probable that the presence of traces of iron on the cathode is due to a secondary electrolytic action, an action which may in time be elucidated and completely avoided.

IX. It is also necessary to remark that, although it is present in such small proportions, the iron deposited on the cathode is present in two different states; by dissolving the deposited metal in weak, warm hydrochloric acid, we can obtain a solution containing traces of iron. On the other hand, there remains a very small black residue, which is not attacked even by concentrated hydrochloric acid, but which is dissolved on boiling and the addition of a little nitric acid. The solution of this black residue in nitric acid gives the reactions of ferric salts. The extremely small quantity I was able to obtain (not more than 0.4 m.grm.) prevented me from studying it any further.

I have finally established that a very small proportion of chromic acid, in an ammoniacal solution of nickel, is sufficient to entirely stop the electrolytic deposition of the nickel, whether iron is present or not.—*Bull. Soc. Chim.*, Series 3, xvii.-xviii., Nos. 18-19.

ON THE ESTIMATION OF THE ORGANIC MATTER IN WATER BY MEANS OF PERMANGANATE OF POTASH.

By FELIX MARBOUTIN and MICHEL FRANCK.

We have estimated the amount of organic matter in solution in various waters around Paris by means of two different processes; the Albert-Lévy method as used in France, and the Forchhammer method used in England. These two methods of estimation give very different results for the same water.

M.grms. of Oxygen Absorbed to Oxidise the Organic Matter (per Litre).

	Albert-Lévy method.	Forchhammer method, modified by Sir E. Frankland.
Seine at Choisy-le-Roi ..	2.92	1.53
" Ivry	3.00	1.66
" Austerlitz	2.60	1.27
" Argenteuil	2.43	1.26
Marne at Neuilly	1.22	0.69
" Saint-Maur	1.13	0.56
Ourcq at the Vilette basin ..	1.46	0.82
Ayre reservoir, Rue Villejust ..	0.24	0.08
Drain at Epinay (Gennevilliers)	1.13	0.39
Drain at Gresillons (Gennevilliers)	1.05	0.36
Drain at Garenne (Achères) ..	0.65	0.24

This table shows that, for river waters, the French method gives figures double those of the English method; while for certain springs and surface drainage waters the numbers are tripled.

In view of this fact, it may be interesting to compare the tables as arranged in England and France for the classification of waters by the estimation of organic matter.

M.grms. of Oxygen Absorbed to Oxidise the Organic Matter (per Litre).

Nature of the water.	FRANCE. Albert-Lévy process.	ENGLAND. Sir E. Frankland by the Forchhammer process.	
	M.grms.	Surface waters from uncultivated land.	Surface waters other than from uncultivated land.
		M.grms.	M.grms.
Very pure ..	Less than 1.0	Less than 1.0	Less than 0.5
Medium purity ..	1.0 to 2.0	1.0 to 3.0	0.5 to 1.5
Suspicious ..	3.0 to 4.0	3.0 to 4.0	1.5 to 2.0
Impure ..	More than 4.0	More than 4.0	More than 2.0

Thus it appears that river waters are classified in the same category both in France and England, in spite of the difference in the results; but such is not always the case with springs and surface drainage water. In France, the Commission on Hygiene did not think it worth while to make any difference between surface waters from uncultivated or cultivated sources, and it is noticeable that they are more strict than in England for springs and surface drainage waters.—*Bull. Soc. Chim.*, Series 3, vols. xvii.-xiii., Nos. 18-19.

NOTE ON A

SOMEWHAT REMARKABLE CASE OF THE RAPID POLYMERISATION OF CHLORAL.

By J. W. MALLET.

A SPECIMEN of anhydrous chloral—trichloroacetaldehyde—of about 250 grms, was contained in a glass vial of a form commonly used in Germany, with small drawn-out neck, hermetically sealed. It had been on hand for more than a year, and had originally been quite clear, but doubtless not absolutely pure, very likely containing traces of sulphuric acid, as a few small white flocks of the polymeric meta-chloral had begun to show at the bottom.

This specimen, with a number of others, had been exhibited on the lecture-table, handled by several persons, and moved back to the collection-room whence it had come. It was placed temporarily on a table, among other bottles, in the afternoon, to be next morning

restored to its place in the collection on the shelves. No one went into this room, which is in a locked portion of the building, before the following day. The temperature was moderate—about 20° or 22° C.—and differed but little from that of the lecture-room. The place on the table occupied by the bottles did not receive any direct rays of the sun.

On coming into the room in the morning a strong odour of chloral at once drew attention to the table, where a circle of half a metre or so in diameter was covered with a thin crust of white meta-chloral. The remains of the sealed vial were found as small fragments scattered about the room, some of them as much as two or two and a half metres away from where the vial had stood, and some of them on the shelves at a higher level than the top of the table, showing clearly that the vial had not fallen down, or been knocked over by a mouse or rat, but had burst, and with some considerable force, although no other bottle standing near it had been broken.

It seems that polymerisation must have taken place so rapidly, and to so large an extent, that the heat evolved in the union of the smaller into larger molecules raised the temperature of the remaining liquid chloral to a point at which the tension of its vapour was more than the vial could withstand.

If this explanation be accepted, the occurrence is in three respects noteworthy. In the first place, the amount of heat evolved in the polymeric change must have been quite large to produce the effect observed, remembering that liquid chloral is only about as volatile as water—has about the same boiling-point under atmospheric pressure; I do not know of any recorded measurements of the thermal value of the change in question. Secondly, the polymeric change must have occurred with a degree of suddenness which is surprising in view of the very gradual transition from liquid chloral to meta-chloral which is usually observed; other specimens, prepared in this laboratory, have taken several years to become entirely solid. Thirdly, it is not easy to imagine what cause can have provoked this sudden polymerisation, there having been less mechanical agitation, less change of temperature, less external disturbance of any kind that can be pointed out, at the time when the change occurred than the specimen had previously been exposed to.—*American Chemical Journal*, xix., No. 9, Nov., 1897.

NOTE ON THE SO-CALLED "SELECTIVE ACTION" OF CYANIDE OF POTASSIUM FOR GOLD.*

By W. A. DIXON.

FOR some years past there has been considerable discussion as to a "selective action" exhibited by very much diluted solutions of cyanide of potassium for gold, so that it passes into the liquid as aurocyanide of potassium, whilst the base metals are left behind in the ore mass during treatment by the cyanide process. This is held to be the reverse of what occurs when a more concentrated solution of cyanide is employed, as in that case a large proportion of base metal in comparison to the gold is obtained in solution. I think the following considerations will show how this occurs, and that there is really no such thing as "selective action." By a selective action it seems to be generally understood that in the weak solution the action is the reverse of what it is in a strong one, an idea of volition on the part of the cyanide being implied in the statement.

The reaction by which gold is dissolved by cyanide of potassium is—



the oxygen being supplied by that present in solution in the water, or by oxidising agents, as chlorine, bromine, ferricyanides, peroxides, &c.; in practice the first is the usual source.

A ton of ore in powder requires from 100 to 110 gallons of water to thoroughly wet it, or, say, an average of 105 gallons, which at 70,000 grains per gallon is equal to 7,350,000 grains. The coefficient of solubility of oxygen at 30 ins. barometric pressure and 60° F. is 0.0295, which, divided by 5 to reduce it to that due to the partial pressure of oxygen in air, gives as the coefficient of absorption of oxygen from air 0.0059, so that 105 gallons of water would contain 43,383 grain measures of oxygen in solution, which would weigh 62 grains. These 62 grains of oxygen contained in 105 gallons, as a maximum, would, in conjunction with cyanide of potassium, dissolve 1527 grains of gold, that is 3 ozs. 3 dwts. 15 grs., and the quantity of cyanide required would be 1001 grains, so that the solution would be very dilute, containing only 0.0047 per cent of cyanogen.

It appears clear to me that we have here a sufficient explanation of the so-called "selective action." Gold in presence of oxygen has in all cases a superior affinity for soluble cyanides, or the soluble cyanides have for gold, than the soluble cyanides have for the compounds of the base metals. These compounds have the same affinity for cyanogen whether oxygen in the free state or easily available is present or not, whilst if oxygen is absent the action on gold is nil. The base metals always occur in gold ores as oxides, sulphides, or other compounds, whilst the gold is in the free state. In these circumstances the gold is first dissolved as long as free oxygen is available, and then, oxygen being exhausted, base metals pass into solution until either they are exhausted or the cyanide is saturated, whichever happens first, a sufficient time being given to complete the reaction. A much diluted solution of cyanide contains much free oxygen in proportion to the cyanide, and therefore dissolves much gold in proportion to the cyanide present, and afterwards little base metal, because there is little cyanide left to saturate, and it therefore appears that the cyanide has selected the gold. In a solution containing more cyanide, gold is dissolved till the oxygen is exhausted, and then the excess of cyanide enters into double decomposition with the compounds of the base metals, which are then found in solution in greater proportion relatively to the gold, and the solution appears to have had a selective action on them.

Some metals in the free state, as zinc, for example, have a superior affinity for cyanogen to gold, but these do not occur free in ores, but when a solution containing gold as cyanide is brought in contact with one of them, as zinc, it dissolves, and the gold is precipitated. Zinc, which is commonly used for this purpose, is moreover known to precipitate gold the more efficiently the more free it is from compounds such as its oxide.

In practical work the oxygen in solution in the water used would be much less than that indicated, as it would be removed by organic and mineral substances undergoing oxidation, but, on the other hand, minerals treated by the cyanide process have usually much less gold than 3 ozs. per ton, or they are treated several times with fresh solutions. This re-treatment is simply a renewal of the conditions favourable to the solution of gold, an oxygen-holding solution of cyanide replacing one which is exhausted of that element.

In the current literature on this subject two distinct sets of reactions have become mixed up, and are spoken of as one, the substances which caused them being called "cyanicides." This word should be confined to those substances which by their action on cyanides decompose them, or set free hydrocyanic acid, which soon decomposes. These substances do not combine with cyanogen

* Read before the Institution of Mining and Metallurgy, Nov. 17th, 1897.

or alkaline cyanides; they kill them, and to such the word cyanide is truly applicable. On the other hand, "cyanide" is not applicable to substances which by entering into combination with cyanogen or cyanides may be rather regarded as causing sleep rather than death, and it is only on these that the so-called "selective action" can be exercised, and the compounds of only three of these are found in gold ores, namely, iron, copper, and zinc. Iron present in the ferric state may be inert or act as a cyanide if present as a salt, *i.e.*, as ferric sulphate; in the ferrous state it combines with cyanide of potassium to form ferrocyanide of potassium, but this also dissolves gold if sufficient oxygen is present, by the reaction—

$6\text{Au} + 2\text{K}_4\text{FeCy}_6 + 4\text{O} + \text{H}_2\text{O} = 6\text{AuKCy}_2 + 2\text{KHO} + \text{Fe}_2\text{O}_3$, and therefore it is not entitled to a position amongst the metals which may be selected. These are therefore reduced to zinc and copper, and the compounds of these are only dissolved in absence of sufficient oxygen with the cyanide of potassium to dissolve the gold. In most gold ores these metals are absent, and then the question of selection does not arise, and solutions of cyanide of any strength may be used, but diluted ones would be most convenient, as they supply the necessary oxygen and consume less cyanide. It is to be supposed that sulphates or other cyanide compounds are removed or decomposed by a preliminary treatment.

In practice it must necessarily happen that solutions containing more than the theoretical quantity of cyanide to the quantity of oxygen present must be used, even when cyanides are absent, in consequence of the gold being distributed in granules throughout an immense mass of ore. A granule of gold exhausts the cyanide and oxygen in its immediate neighbourhood, and action then ceases until a fresh supply of both arrives by diffusion from the surrounding solution. I do not know that the relative rate of diffusion of oxygen and cyanide in watery solution has been determined, but it must be that 16 of oxygen will diffuse sooner than 260 parts of cyanide of potassium, and therefore an excess of cyanide must be used to compensate for this difference.

SOME NEW DERIVATIVES OF DIACETYL.

By HARRY F. KELLER and PHILIP MAAS.

SINCE communicating the results obtained in studying the action of oxidising agents upon diacetyl, we have continued our work on this diketone in several directions.

The ready and quantitative conversion of diacetyl into acetic acid by certain oxidising agents, notably hydrogen peroxide, induced us to try whether the halogen-substituted products, dibromo-diacetyl and tetrabromo-diacetyl, would behave in an analogous manner. If they, respectively, yielded bromacetic acid and dibromacetic acid, their symmetrical structure might be regarded as definitely established. Thus:—



and—



While both compounds were found to be readily attacked by the oxidising agent, only the dibromo-derivative gave something like the expected result.

A weighed quantity of this substance was treated in the cold with a moderate excess of hydrogen peroxide solution. The greasy scales gradually disappeared, and the liquid became strongly acid. Upon evaporating, it gave off hydrobromic acid, and left a residue consisting of a mixture of bromacetic and glycollic (oxyacetic) acids. This mixture was completely converted into the latter acid by prolonged boiling with water, according to the equation—



The characteristic calcium salt was prepared. It was obtained in stellar aggregations of needles, having a silky lustre, insoluble in alcohol and only sparingly soluble in cold water. It contained:—

	I.	II.	Calculated for (C ₂ H ₃ O ₂) ₂ Ca + 4H ₂ O.
Ca	15.06	15.54	15.26
H ₂ O	26.97	26.47	27.48

The salt is understood to be somewhat efflorescent, and the percentage of water therefore a little too low. The dehydrated salt gave Ca = 21.32 per cent, instead of 21.05 per cent required by theory.

The blue copper salt was also obtained.

A mixture of several acids is produced when dibromo-diacetyl is heated with nitric acid. We have not been able to effect their separation, but have reasons to believe that oxalic acid and monobromacetic acid are amongst them.

When tetrabromodiacetyl was treated with hydrogen peroxide, its yellow colour disappeared slowly. A white flocculent precipitate formed while the dense powder dissolved. The colourless solution had a peculiar penetrating odour, and deposited, upon standing, another crop of the product in the form of long colourless needles with a silky lustre. From 3 to 4 grms. of this substance were thus obtained from 10 grms. of the diketone.

The mother liquor, still having the characteristic odour of the crystallised product, was extracted with ether, but the latter, on evaporating, left only a small quantity of an oily liquid.

The crystals were found to be insoluble in water, and very freely soluble in alcohol, ether, benzene, glacial acetic acid, and other organic solvents. From an ethereal solution very beautiful prismatic crystals, more than a centimetre long, were obtained. They belong, without doubt, to the rhombic system, and consist of several prisms, the orthopinacoid and a brachydome. From the solution in acetic acid the substance is re-precipitated in the form of long fine needles when much water is added. It melts at 72.8°, and at higher temperatures sublimes, apparently without decomposition.

Analyses of the carefully purified substance yielded:—

	I.	II.	Calculated for C ₂ HBr ₂ O. Per cent.
C	8.13	8.04	7.94
H	0.56	0.26	0.22
Br	87.99	88.04	88.30
O	[3.32]	[3.66]	3.53

Determinations of the molecular weight by the cryoscopic method, by means of Beckmann's apparatus and glacial acetic acid as the solvent, gave:—

	I.	
Used solvent	19.485 grms.	
Substance	0.1788 grm.	
Depression	0.084°	

	II.	
Solvent	19.663 grms.	
Substance	0.3360 grm.	
Depression	0.146°	

I.	II.	Calculated.
461	435	452

The composition and molecular weight correspond to the formula C₂HBr₂O, which is that of *pentabromacetone*. That this is really the compound we obtained is further proved by a comparison of the physical properties* with those observed by other chemists, as well as its behaviour

* The melting-point is given by Beilstein (3rd ed., p. 989) as 70°. Although we repeated the determination several times, using material purified with scrupulous care, and the best thermometers, it invariably gave the same result, 72.8°.

upon boiling with barium hydroxide; bromoform was produced and was identified, after distilling it in a current of steam, by its odour and boiling-point, and the formation of phenylcarbylamine when the alcoholic solution was treated with aniline and caustic potash.

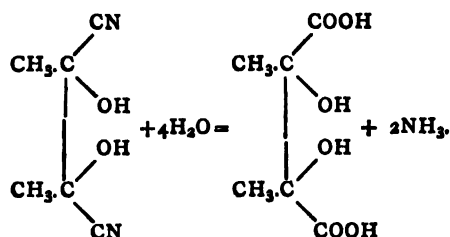
It is remarkable that an acetone derivative should result from the oxidation of a diketone, for methyl-ethyl ketone can be converted by oxidation into diacetyl (Fileti and Ponzio, *Gazz. Chim. Ital.*, xxv., 233).

The oily product mentioned above, and which was extracted from the solution after the pentabromacetone had separated, may have been tribromacetone. It had a powerful odour, decomposed upon heating, and did not respond to the carbylamine reaction.

Attempts to oxidise tetrabromodiacetyl with nitric acid have not as yet given any definite results. Although the substance dissolves rapidly on warming in the concentrated, especially the red fuming, acid, the greater part of it separates again on cooling or diluting with water in large yellow crystals.

Cyanohydrins.—Another class of diacetyl derivatives to which we have lately given some attention are the cyanohydrins.

Fitting and one of us have shown that diacetyl and hydrocyanic acids unite quantitatively to form a dicyanohydrin, and that this may be readily converted into dimethylracemic acid.



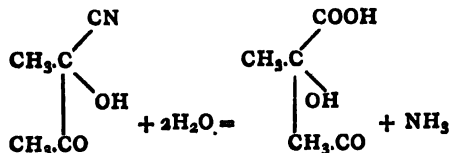
Our recent experiments were undertaken—

1. To attempt the preparation of a monocyanohydrin of diacetyl.

2. To produce halogen-substituted cyanohydrins.

If we succeeded in converting the latter into the corresponding acids, we would have the first derivatives of tartaric acid in which hydrogen is replaced by halogen.

Diacetyl and hydrocyanic acid (30 per cent solution) were brought together in molecular proportions. A rise of temperature and the vanishing of the yellow colour indicated that combination was taking place. The liquid was gently heated; and then allowed to stand for some time in a warm place. After extracting with ether, and evaporating this solvent, there remained a colourless viscous liquid. This was placed over dehydrating materials, and repeatedly cooled to very low temperatures without showing any signs of crystallising or congealing. It was doubtless the expected compound, though probably very impure. We now attempted to produce the corresponding acid—



by treatment with strong hydrochloric acid. This was tried in a number of ways; but although even the most concentrated acid (saturated at 0°) had no appreciable effect at ordinary temperatures, the slightest warming destroyed the compound, with formation of dark resinous products, from which an acid of definite composition could not be isolated.

More encouraging were the results of our experiments

upon dibromodiacetyl. It was found to combine with hydrocyanic acid quite readily, and yielded a well-crystallised dicyanohydrin. From the ethereal extract it was obtained in fine colourless crystals, having a strong lustre, and melting with decomposition at 177°.

Its composition is established by these determinations:—

	Found.		Calculated.
	I. Per cent.	II. Per cent.	Per cent.
N	9.38		9.39
Br	54.35	54.74	53.69

The excess of bromine is probably due to the formation of calcium cyanide when the substance is ignited with lime.

Experiments are in progress to transform the nitrile into the acid; the amide of the acid has been obtained in small quantities.

We have also found that tetrabromodiacetyl can be made to combine without difficulty with two molecules of hydrocyanic acid. The resulting body seems to be analogous to the tetrachlorodiacetyl dicyanohydrin described by Levy and Witte (*Ann.*, ccliv, 98). As yet, however, we have not prepared a sufficient quantity to warrant the publication of definite data.

Dichlorodiacetyl.—As Levy and his pupils (*Ann.*, ccxlix., 93) obtained their tetrachlorodiacetyl by a complicated process, the action of potassium chlorate and hydrochloric acid on chloranilic acid, we have tried to prepare it by direct chlorination of diacetyl. It was found that at first the action of chlorine upon the diketone, dissolved in carbon disulphide or chloroform, is quite energetic, but the only product we have so far obtained proves to be dichlorodiacetyl. It closely resembles the dibromo-derivative; soluble in carbon disulphide, chloroform, boiling petroleum ether, and warm benzene, it crystallises in yellowish unctuous scales melting at 124.5°. A chlorine determination gave—

	Found.	Required for C ₂ H ₂ Cl ₂ O.
	Per cent.	Per cent.
Cl	45.63	45.80

—*Journal of the Franklin Institute*, vol. cxliv., No. 5,

A CONTRIBUTION TO THE STUDY OF OXYGEN AT LOW PRESSURES.*

By R. THRELFALL, M.A., Professor of Physics in the
University of Sydney, and FLORENCE MARTIN.

WHEN a mass of oxygen is enclosed in a tube and the mercury pressure on it continuously diminished, it is found that at about 0.7 m.m., and over a certain range of lower pressures, the gas appears to undergo a change of condition. The phenomenon may be described in the words of Bohr, its discoverer (*Wied. Ann.*, xxvii., p. 475), "A given mass of oxygen is enclosed in a tube, and the mercury adjusted so as to give to a pressure rather less than 0.7 m.m. If the volume of the gas is now reduced by raising the pressure, say to 0.8 m.m., it is noted that this pressure will not remain constant, but varies more or less with lapse of time. In three to five hours the pressure will fall by some 12 per cent of its initial value (the volume being constant). After five hours the pressure was found to have attained its steady value, so far as observations extending over twenty-four to thirty-six hours could determine."

This curious behaviour of oxygen was also noted by Baly and Ramsay (*Phil. Mag.*, xxxviii., p. 324, 1894), who observed that at a pressure of about 0.75 m.m. oxygen becomes unstable as to its pressure-volume relation, and

* Read before the Royal Society of N. S. Wales, June 2, 1897.

that the equilibrium condition is not attained until after seventy-eight hours rest. The slightest change of pressure or volume then upsets the equilibrium, and time has again to elapse before a steady state is attained. It appears likely either that the oxygen forms an allotropic modification or that it forms some compound with mercury or other material present and with which it is in contact.

It will be noted that, according to Bohr, the volume of the gas tends to increase below 0.7 m.m., indicating that the molecules of oxygen are partly split up. In this case, therefore, it would be reasonable to infer an increase of oxidising power, and it is possibly to this cause that the soiling of the fall-tubes of Sprengel pumps is to be attributed. It appears worth while, therefore, to try to arrange some chemical test capable of showing the presence of active oxygen.

The two following test solutions were found to satisfy the conditions, though one was more sensitive than the other. One condition of course is that the test solution must not have a vapour-pressure comparable with 0.7 m.m. The first indicator tried was a solution of indigo in pure sulphuric acid. This is bleached by ozone, but experiment showed that the reaction does not afford a very delicate test of the presence of that gas. Another solution was therefore tried, consisting of potassium iodide and starch dissolved in glycerin. The glycerin was carefully dried at a temperature of 260° C. When cool, some of it was mixed with a small quantity of powdered potassium iodide. A very small quantity of starch was added to the remainder of the glycerin, which was then slowly heated till the starch was quite dissolved and the liquid again became transparent. When cold, this portion was mixed with the potassium iodide solution—a solution so prepared is not affected by ordinary oxygen, but one bubble of the gas which has passed through an ozone tube turns it bright yellow, and three bubbles give it a dark blue, almost black colour. This seemed sufficiently sensitive, and was accordingly adopted. Of course the starch is not absolutely necessary, iodine being liberated in large enough quantities to colour the solution, but it was considered to be of some advantage to use it as an additional verification. Oxygen was prepared and purified in the usual manner, and stored in a gas-holder.

On leaving the gas-holder the gas passed through (1) a system of purifying tubes containing (a) nitrate of silver, (b) solid potash, (c) sulphuric acid, (d) phosphorus pentoxide; (2) a wash-bottle containing a small quantity of the potassium iodide solution; (3) an ozoniser by which the oxygen could, when required, be ozonised without altering any of the apparatus.

Two diagonal glass taps, in series, allowed the purified gas to pass into the exhausted part of the apparatus. This consisted of a glass tube about 0.2 c.m. in diameter and 30 c.m. long, to which was fixed a mercury pressure gauge of the U type, 0.1 c.m. in diameter. In order to prevent a possible loss of active oxygen through the action of the mercury in the gauge, the latter was connected to the exhausted space by a capillary connection.

The exhausted tube was connected through a small wash-bottle with a Fleuss pump, the wash-bottle containing a small quantity of the sensitive solution. A similar wash-bottle, containing the same solution, was arranged to stand close to the bottle through which the gas was passed in order to enable colour comparisons to be made. All the apparatus was, practically speaking, either fused together or had joints protected by paraffin and mercury, the use of indiarubber being of course inadmissible. The Fleuss pump was worked by an electric motor, and the taps were adjusted until a steady stream of oxygen could be passed through the apparatus at a pressure of about 0.25 m.m. of mercury.

The apparatus, after having been made entirely airtight, was filled with oxygen and exhausted several times; a steady stream of oxygen, at atmospheric pressure, was then run through it for an hour, in order to get rid of

traces of air. It was then exhausted and kept at a constant pressure of nearly 0.25 m.m. (never less than 0.1 nor greater than 0.4 m.m.), with the oxygen bubbles coming through at the rate of twenty per minute. Each bubble of oxygen, on reaching the exhausted tube, was therefore reduced in pressure over the range of instability. After six hours, no change having taken place in the potassium iodide solution, the apparatus was filled with oxygen at atmospheric pressure and left for several hours. This experiment was repeated during three days; that is to say, the oxygen was passing through the apparatus at a pressure of 0.25 m.m. for 17.5 hours altogether. At the end of this time, no trace of the ozone reaction being observable, it was considered advisable to ascertain whether if a very small proportion of the oxygen passing through had become converted into ozone, so minute a quantity, at so low a pressure, would affect the test solution. With this object, the wires of the ozoniser were now joined up, and it was found that in one minute a faint yellow colouring of the solution, slight but distinctly visible, occurred. Evidently, therefore, twenty bubbles of electrically ozonised oxygen produce more effect than 21,000 bubbles of oxygen which has been simply subjected to the effects of low pressure. And even if the experiment described above is not considered to prove, with sufficient conclusiveness, that low pressure alone has no power to cause the formation of ozone in oxygen, it must at least be admitted that the ozone so formed is less than 1/1000th of the quantity produced by an ozoniser in the ordinary way in the same volume of oxygen, and as this can scarcely exceed 5 per cent of the whole volume, the ozone formed by lowering the pressure cannot be so much as 0.005 per cent of the volume of oxygen present.

We must not neglect to state that our curiosity in this matter was stimulated to the experimenting point by a letter from our friend, Mr. W. Sutherland, of Melbourne, who considered, on grounds based on the kinetic theory of gases, that allotropic oxygen of some kind would most likely be found at about the pressure we employed. The soiling of Sprengel pumps, however, as well as the experiments of Baly and Ramsay, had previously led us, independently, to infer the possibility of a production of active oxygen under the conditions we have mentioned.

A REVISION OF THE ATOMIC WEIGHT OF NICKEL.*

FIRST PAPER.—THE ANALYSIS OF NICKELOUS BROMIDE.

By THEODORE WILLIAM RICHARDS

and

ALLERTON SEWARD CUSHMAN.

Introduction.

OF the atomic weights to-day those of nickel and cobalt are among the most interesting, not only because the two values are so close together, but also because the purely elementary character of these metals has been recently doubted (*Zeit. Anorg. Chem.*, ii., 235; *Berichte*, 1889, xi., 2026). A careful study of the available literature upon the subject leaves the impartial critic in grave doubt as to the true values to be accepted, hence an experimental revision seems to be imperative; and the present work is a part of a comprehensive attempt to conduct such a revision. The very useful and complete index of the work which has been done upon this subject, recently published by Professor Clarke (*"Recalculation of the Atomic Weights," Smithsonian. Misc. Coll., Constants of Nature, Part V., 1897, p. 297*), makes a detailed statement of the various researches unnecessary here; but a chronological list is appended.

* Contribution from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, vol. xxxiii., No. 7.

The Atomic Weight of Nickel.
O = 16.

1826.	By Analysis of Chloride.	
	Rothoff	59.1
1852.	Reduction of the Oxide by Hydrogen.	
	Erdmann and Marchand	58.2—58.6
1856.	Conversion of Nickel to Sulphate.	
	Deville	58.85
1857.	Analysis of the Oxalate.	
	Schneider	58.07
1858.	Analysis of the Sulphate and Chloride.	
	Marignac	58.4—59.29
1860.	Synthesis of the Chloride.	
	Dumas	59.02
1863.	Reduction of the Oxides in Hydrogen.	
	Russell	58.74
1866.	Analysis of Nickel Potassic Sulphate.	
	Sommaruga	58.03
1867.	By relation to Gold, out of Sod. Auro-Chloride.	
	Winkler	59.45
1867.	Measurement of Hydrogen evolved by acting on Nickel with HCl.	
	Russell	58.77
1871.	Analysis of Strychnine and Brucine Nickel-cyanides.	
	Lee	58.01
1883.	Analysis of the Sulphate.	
	Baubigny	58.73
1886.	Reduction of Oxide (NiO) by Hydrogen.	
	Zimmermann	58.71
1890.	Mond, Langer, and Quincke	58.58
1892.	Conversion of Sulphate to Oxide, and Reduction in Hydrogen.	
	Schützenberger	58.54
1892.	Krüss and Schmidt	57—64
1893-94.	Analysis of Nickelous Chloride and by relation to Iodine.	
	Winkler	59.05—58.87
	Value accepted by Ostwald, 1890.. Ni = 58.5 (<i>Lehrbuch</i> , i., p. 96).	
	Value accepted by Clarke, 1896 .. Ni = 58.687 ("Recalculation of the Atomic Weights," <i>loc. cit.</i>).	
	Value accepted by Seubert, 1896 .. Ni = 58.9 (<i>Zeit. Anorg. Chem.</i> , xiii., 229).	

by securing unanimous results from a number of compounds and methods, it is equally obvious that a single method, well worked out, is far better than twenty incomplete ones; hence the present work was confined for the present to a single compound.

In our choice of material, we were guided by experience already gained in this Laboratory. The advantage of the bromides as typical compounds in atomic weight determinations has been discussed by one of us in preceding publications, and requires no further mention. The rather meagre current descriptions of the properties of anhydrous nickel bromide did not seem encouraging, but the progress of the research showed that it is possible to dry this salt, weigh it, and dissolve it in water in a perfectly normal condition. Since this was the case, nickelous bromide was naturally chosen as our starting point.

The Preparation and Properties of Nickelous Bromide.

Finely divided nickel, when heated to a red heat in a stream of dry bromine vapour, readily forms the bromide, which sublimes at bright redness. The colour of the sublimate varies from a pale straw-yellow to a dark bronze-brown, according to the state of aggregation. At a red heat in the presence of traces of air or moisture, the salt loses traces of bromine with the formation of bright green nickelous oxide, unless much hydrobromic acid is present. We have never obtained evidence that an oxy-bromide is formed under these conditions.

The sublimed bromide is almost insoluble in cold water, but solution soon becomes apparent to the eye in water at 50°. In water at 90° the salt dissolves less slowly, but a grm. still requires an hour or two for its complete solution. When originally free from oxide, the sublimed bromide dissolves in water even at the boiling point to form a solution of perfect clearness. According to Berthelot (*Anal. Chim. Phys.* [2], xliv., 389) a solution of nickelous bromide left for some time in contact with air deposits some flakes of the oxide. We have never met with the slightest evidence of the truth of this statement in the case of a dilute solution. If, however, the nickelous bromide contained only a slight admixture of the oxide, this oxide might escape observation until it had settled out upon the bottom of the vessel. Dilute nitric acid does not materially hasten the solution of the bromide. The sublimed crystalline salt is hygroscopic in character, although not nearly to the extent which we had been led to expect. From several experiments carried out to test

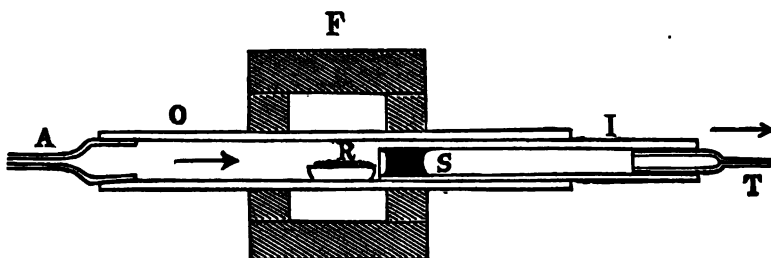


FIG. 1.—SECTION OF APPARATUS FOR SUBLIMATION.
A, Glass tube for admitting bromine vapour. O, Outer porcelain tube. I, Inner porcelain tube. T, Glass outlet tube.
B, Perforated Fletcher furnace. C, Boat containing nickel. S, Sublimed bromide.

A glance at this list shows the lack of consistency in the results obtained. In many cases this is sufficiently accounted for by the inadequacy of the methods and the known impurity of the materials used, without taking into consideration the unconfirmed admixture of unknown impurities. A critical investigation of the data already published would be an endless and perhaps unprofitable labour, and we prefer at this time rather to record the method and results of our own work than to enter into any discussion of claims heretofore advanced.

While it is obvious that certainty is to be obtained only

this point it appeared that freshly sublimed nickelous bromide exposed to the free air of the room absorbed about 0.1 milligrm. per grm. in ten minutes.

Since the value of the selection of nickelous bromide as the starting point in the determination of the atomic weight of nickel depends on the possibility of dissolving the salt in hot water without the slightest loss of bromine, it seemed necessary to investigate this point. To this end, a mass of several grms. of the sublimed compound was suspended in a flask arranged in such a way that the atmosphere of the flask during the solution could be

swept through a bulb-tube containing a mixture of potassium iodide and starch. Any formation of nickelous oxide or oxybromide during the heating of the solution could have taken place only by disengagement of bromine, which would have been shown by the blueing of the indicator in the bulb. Not the slightest tinge of blue colour appeared at any time during the experiment, in spite of the fact that the solution of nickel bromide was finally subjected to prolonged boiling. That the iodo-starch mixture was exceedingly sensitive to very minute traces of free bromine was assured by experiment in the first place. This experiment is detailed here, however, merely as corroborative testimony, and is not advanced as being final on the subject; the best evidence is yielded by the quantitative results of our series of analyses.

Being now sure that we could analyse the salt if we could obtain it in a pure state, we next proceeded to determine whether it could be prepared in a condition altogether suitable for weighing and analysis. The first sublimations were performed in an ordinary combustion tube in an atmosphere of carbon dioxide. These sublimations yielded in no case material that was above suspicion, and none of it was used in our analyses, the experiments being carried out merely as a study of the properties of the substance under examination. Since it seemed possible that the carbon dioxide used might suffer partial dissociation at the high temperature necessary for the sublimation, nitrogen was substituted for this gas. After many experiments with nitrogen, both alone and when mixed with bromine vapour, it was found best to carry on the sublimation of the nickelous bromide in a stream of nitrogen mixed with hydrobromic acid. The elaborate apparatus constructed by Mr. G. P. Baxter for supplying suitable mixtures of any or all of these gases and vapours for preparing and subliming the salt will be described in a later paper, upon the atomic weight of cobalt.

The temperature at which the salt sublimes lies not far from that at which the hardest glass begins to soften, hence we found it advantageous to conduct the sublimation in porcelain. In order to collect the product a small porcelain tube, used as the receiver, was fitted, telescope fashion, inside of the larger tube containing the substance to be vaporised. In this way the pure crystals could be removed without contamination. Since nickelous bromide is decomposed either by oxygen or by water at a red heat, unless a very large excess of hydrobromic acid gas is present, both oxygen and water must be excluded with scrupulous care during its final sublimation. At first we were not able to accomplish this complete exclusion, so that most of the bromide used in the preliminary series of analyses contained traces of green crystalline oxide, which were carefully collected and weighed. In making the final calculation, the weight of this oxide was subtracted from the total, in order to obtain the weight of the bromide. Subsequently, with improved apparatus, the bromide was obtained in a state of satisfactory purity; it gave a perfectly clear solution in water, leaving nothing to be desired. With the improved arrangement the oxide itself was quickly converted at a dull red heat into the bromide, by means of a stream of hydrobromic acid gas. Hence it was possible to purify nickelous bromide contaminated with oxide by simply treating it for a few minutes in this fashion; in practice, the scheme worked very well.

Chemical literature contains no satisfactory determination of the specific gravity of the anhydrous salt; hence, in order to reduce our weighings to the vacuum standard, the following determination was made:—3.3196 grms. of pure dry nickelous bromide displaced upon one occasion 0.6162 grm. and upon another 0.6156 grm. of redified toluol at 28°. Since the specific gravity of the toluol at this temperature was 0.860, referred to water at the same temperature, the specific gravity of the bromide must be 4.64. Nickelous bromide is insoluble in toluol.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, November 4th, 1897.

(Concluded from p. 276).

114. "On some Yellow Vegetable Colouring Matters." By A. G. PERKIN.

The *Rhus rhodantha*, a tree growing to the height of 70 or 80 feet, is indigenous to northern New South Wales. The colouring matter $C_{15}H_{10}O_6$ is identical with fisetin. A glucoside of fisetin, $C_{36}H_{30}O_{16}$ ($C=60.18$; $H=4.45$), colourless needles, m. p. 215–217°, is also present; it is decomposed with difficulty by boiling dilute acids. This closely resembles fustin, $C_{38}H_{26}O_{23}$ or $C_{36}H_{26}O_{14}$ ($C=63.34$; $H=3.81$), m. p. 217–219°, the fisetin glucoside of *R. Cotinus* (Schmid, *Ber.*, 1886, xix., 1753), but differs from it in percentage composition. Its decomposition with acid would be closely expressed by the equation $C_{36}H_{30}O_{16} + 2H_2O = 2C_{15}H_{10}O_6 + C_6H_{14}O_6$, if rhamnose or glucose are liberated by this reaction. Gallic acid was also isolated, evidently as a decomposition product of gallotannic acid contained in the wood.

Berberis ortuensis, a plant resembling *Berberis vulgaris*, flourishes in Cyprus. It was found to contain berberine, but no colouring matter of the mordant yellow class.

The perianths surrounding the seeds of *Rumex obtusifolius* contain a trace of quercetin, which is interesting, as in many roots of this species methylanthraquinone derivatives also exist. It is also pointed out that the leaves and green stems of madder (*Rubia tinctoria*) contain a yellow colouring matter which will be examined.

115. "Naphthylureas." By GEORGE YOUNG, Ph.D., and ERNEST CLARK.

The mononaphthylureas may be prepared by the action of potassium cyanate on the hydrochloride of the corresponding naphthylamine. In consequence of the rapid conversion of the mononaphthylureas into the symmetrical dinaphthylureas which takes place on heating, even below the melting-points of the former, the true melting-points have escaped the observation of previous authors. α -Naphthylurea melts at 213–214°, at which temperature it is converted into di- α -naphthylurea, melting at 284–286°. β -Naphthylurea melts at 213–215°, and immediately forms di- β -naphthylurea, melting at 289–290°. Acetyl- α -naphthylurea, m. p. 214–215°; benzoyl- α -naphthylurea, m. p. 243–245°; acetyl- β -naphthylurea, m. p. 202–203.5°; benzoyl- β -naphthylurea m. p. 219–220°.

116. "Benzoylphenylsemicarbazide." Preliminary Notice. By GEORGE YOUNG, Ph.D., and HENRY ANNABLE.

In a previous communication presented to the Society (*Trans.*, 1897, lxxi., 200), attention was drawn to the disagreement between the melting-points of benzoylphenylsemicarbazide, 202–203°, as observed by Michaelis and Schmidt (*Ber.*, 1887, xx., 1713), and 210–211° as observed by Widman (*Ber.*, 1893, xxvi., 945). It described the preparation and examination of this substance—melting at 202–203°—and suggested the possible existence of two benzoylphenylsemicarbazides both having the constitutional formula $C_6H_5N(COC_6H_5) \cdot NH \cdot CO \cdot NH_2$. Shortly after the publication of this paper, Dr. Widman had the courtesy to submit a sample of his preparation for comparison. This sample had been observed by Dr. Widman to melt at 210–212°; the authors found it to melt at 211–212°. Their thermometer agreed therefore with Dr. Widman's. A comparison of the properties of the two preparations led to exceedingly interesting results. Widman's benzoylphenylsemicarbazide seemed to be almost, if not quite, insoluble in boiling benzene, the melting-point remaining unaffected. The author's benzoylphenylsemicarbazide was fairly soluble in boiling benzene, crystallising out again on cooling. Mere re-

crystallisation from benzene did not affect the substance, but prolonged boiling with benzene caused a gradual rise of the melting-point. On the other hand, the substance was easily soluble in boiling water and crystallised out on cooling unchanged, whereas Dr. Widman's preparation dissolved in boiling water with difficulty and crystallised out on cooling, with the melting-point considerably lowered. These results induced the authors to undertake a thorough investigation of the formation and properties of benzoylphenylsemicarbazide. They have been able to determine that the action of benzoyl chloride on phenylsemicarbazide produces under different conditions three distinct forms of benzoylphenylsemicarbazide. These three forms melt respectively at 202–203°, 205–206°, and 210–211°. They are each capable of conversion into either of the other two. They exhibit different and characteristic crystalline structures under the microscope. They possess different solubilities and densities. The form of highest melting-point seems incapable of solution without undergoing at least partial change into one or other of the lower melting forms, but pure solutions of these latter may be easily prepared. These solutions have no action on polarised light. The authors are at present engaged in examining the physical properties of these substances and in extending the investigation to a number of other closely related compounds, in the hope of being able to determine whether they are capable of existence in two or more modifications.

117. "Sulphocamphylic Acid." By W. H. PERKIN, jun.

In a previous communication (*Proc.*, 1895, xi., 23) it was shown that when the potassium salt of sulphocamphylic acid is treated with phosphorus pentabromide, the sulphobromide, $C_8H_{12}(SO_2Br)CO_2H$, is produced, and from this substance by elimination of sulphur dioxide an acid of the formula $C_8H_{12}BrCO_2H$ was obtained, which, as it gives β -camphylic acid, $C_8H_{12}CO_2H$, on treatment with alcoholic potash, may be called *bromo-dihydro- β -camphylic acid*.

During the course of further experiments, the corresponding *camphylic sulphochloride*, $C_8H_{12}(SO_2Cl)CO_2H$, has been obtained by treating the potassium salt of sulphocamphylic acid at 0° with phosphorus pentachloride. This substance melts at 168–170°, and at the same time slowly undergoes decomposition with evolution of sulphur dioxide and formation of *chlorodihydro- β -camphylic acid*, a crystalline substance which melts at 105–106°.

Like the corresponding bromo-compound, it is decomposed by boiling with alcoholic potash, with elimination of hydrogen chloride and formation of β -camphylic acid.

In the last communication on sulphocamphylic acid (*Proc.*, 1896, xii., 189) it was stated that, when β -camphylic acid was treated with phosphorus trichloride, and the product distilled under reduced pressure, the chloride of an acid melting at 130° is obtained which was called *iso- β -camphylic acid*, because at the time it was thought that this acid might prove to be isomeric with β -camphylic acid. It has since been found that the reaction does not proceed in this way, but that the following much more remarkable change takes place. When the chloride of β -camphylic acid is distilled, there is, as already mentioned, some decomposition and charring, and during this distillation the chloride is reduced almost completely to the chloride of an acid, $C_8H_{14}O_2$, which on investigation has been found to be identical with isolaunonic acid, the acid which Koenigs and Hoerlin (*Ber.*, 1893, xxvi., 813), and the author (*Proc.*, 1893, ix., 109) obtained by the elimination of sulphuric acid from sulphocamphylic acid.

This same isolaunonic acid (together with a liquid acid, which is possibly an isomeride) is obtained when β -camphylic acid is reduced with sodium amalgam under certain conditions, and quite lately it has also been obtained in large quantity by fusing sulphocamphylic acid with soda in a cast-iron pot.

When fused in a nickel dish with caustic soda, sulpho-

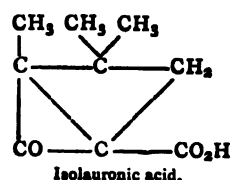
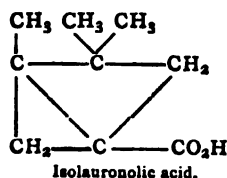
camphylic acid yields a mixture of α - and β -camphylic acids, $C_8H_{12}O_2$, but when an iron pot is used, the iron acts as a reducing agent, and the product, which is found to contain quantities of ferric oxide, on treatment in the usual way yields large quantities of isolaunonic acid, $C_8H_{14}O_2$.

Isolaunonic acid is, as Koenigs and Meyer (*Ber.*, 1894, xxvii., 3466) showed, readily oxidised to isolaunonic acid, $C_8H_{12}O_3$, a ketonic acid which gives a well characterised oxime and a semicarbazide. On reduction with sodium amalgam, the author finds that isolaunonic acid is readily converted into *dihydroisolaunonic acid*, $C_8H_{14}O_3$ (m. p. 88°), a result differing somewhat from that of Koenigs and Meyer, who obtained in this way a lactone, $C_9H_{14}O_2$, melting at 47–50°, together with a substance melting at 80–81°, which they consider to be a mixture of two acids, $C_8H_{14}O_3$ and $C_8H_{16}O_3$.

The author has further studied the action of oxidising agents on isolaunonic acid, and finds that, under certain conditions, this acid is split up into dimethylsuccinic acid, $COOH \cdot C(CH_3)_2 \cdot CH_2 \cdot COOH$ and a ketonic acid, $C_8H_{14}O_3$, which melts at 51°.

This ketonic acid on oxidation is converted into *adamethylglutaric acid*, $CO_2H \cdot C(CH_3)_2 \cdot CH_2 \cdot CH_2 \cdot CO_2H$, and it therefore evidently has the constitution $CH_3 \cdot CO \cdot C(CH_3)_2 \cdot CH_2 \cdot CH_2 \cdot CO_2H$, and is identical with the acid previously obtained (*Proc.*, 1896, xii., 190), by oxidising β -camphylic acid.

A careful study of the results obtained in this long series of experiments on sulphocamphylic acid and the acids derived from it, seems to the author to clearly indicate that the constitutions of isolaunonic and isolaunonic acids are most probably represented by the following formulae:—



As sulphocamphylic acid on heating is resolved into isolaunonic acid and sulphuric acid, and on the other hand, isolaunonic acid, as was indicated in a previous communication (*Proc.*, 1893, ix., 109) and has since been proved, when heated with sulphuric acid at 90°, is again converted into sulphocamphylic acid, it follows that the determination of the constitution of isolaunonic acid will throw most important light on the formula of sulphocamphylic acid, and on the remarkable changes which take place during the formation of this sulpho-acid from camphoric acid.

The discussion of these points and of their bearing on the constitution of camphoric acid, the author must reserve for a detailed description of his experiments, which he hopes soon to be able to lay before the Society.

ROYAL INSTITUTION.

General Monthly Meeting, December 6th, 1897.

Sir JAMES CRICHTON-BROWNE, M.D., F.R.S., Treasurer and Vice-President, in the Chair.

THE following were elected Members:—The Hon. H. M. Birdwood, C.S.I.; Major John Leslie; Capt. H. G. Lyons, R.E.; and Mr. Cecil Powney.

The special thanks of the Members were returned to Professor Dewar, LL.D., F.R.S., for his present of a portrait of Mr. Benjamin Vincent, Honorary Librarian of the Royal Institution.

NOTICES OF BOOKS.

Handbook (A) for Chemists of Beet-sugar Houses and Seed-culture Farms. Containing Selected Methods of Analysis, Sugar-house Control, Reference Tables, &c. By GULFORD L. SPENCER, D.Sc., of the U.S. Department of Agriculture. New York: John Wiley and Sons. London: Chapman and Hall, Lim. 1897. Pp. x.—475. 18mo., Limp Morocco Tuck.

It is interesting to note that such great advance has been made in the beet-sugar industry in the United States that already there is an opening for a book devoted exclusively to the sugar-beet. Less than ten years ago, when Dr. Spencer published his "Handbook for Sugar Manufacturers," the manufacture of sugar was confined to that from cane cultivated in the Southern States; sorghum was attracting some attention, but had been nearly abandoned. At present the beet-sugar industry bids fair to attain enormous proportions in America.

The "Handbook" contains, in a compact and attractive form, a fund of information on the chemistry and treatment of sugar-beets that cannot be found elsewhere without access to an extensive library. It treats in sections of sugar-house control; sugar-analysis by optical methods and by chemical methods; sampling and averaging, analysis of the beet, juice, syrup, and molasses, filter press-cake, and residues of divers kinds; analysis of saccharates, bone-black, chimney-gases, as well as of the crude materials used, such as limestone, sulphur, coke, and water; seed selection, seed testing, and special reagents have their share of space. Seventy pages of useful tables are followed by one hundred and twenty-five pages of blank forms for practical use in sugar-house work, besides thirty pages for reporting yield and losses.

The illustrations illustrate the text, and the type, though small, is very clear. Full-face type heads each paragraph.

The volume contains an index.

H. C. B.

CORRESPONDENCE.

CONVERSION OF THERMOMETRIC SCALES.

To the Editor of the Chemical News.

SIR,—In the *Meteorologische Zeitschrift* for the month of October, G. Hellmann (see also *Revue Scientifique*, November 20, 1897, p. 663), there is an ingenious formula for the conversion of Fahrenheit degrees into those of Centigrade, which may interest you if you have not seen it. The usual formula is, of course,—

$$\frac{(F^{\circ} - 32) \times 5}{9} = C^{\circ}.$$

G. Hellmann:—

$$\frac{5}{9} = 0.5555 = \frac{1.111}{2} = \frac{1}{2} + \frac{0.1}{2} + \frac{0.01}{2} + \dots$$

Let 88 be the number of degrees Fahrenheit, we have then—

$$\begin{array}{r} 88 - 32 = \frac{56}{2} = 28. \\ 28 \\ 2.8 \\ 0.28 \\ \hline 31.08 C^{\circ} \end{array}$$

I would venture to point out that Reamur's scale may be converted into that of Celsius in a somewhat similar manner.

$$\frac{4}{9} = 0.4444 \times 2.5 = 1.0 = 2.5 \times 4 = 1.0,$$

$$\left(\frac{F - 32}{10}\right) \times 4 = R^{\circ}.$$

Example:—

$$\begin{array}{r} \frac{212 F - 32}{10} = 18 \times 4 = 72.00 \\ 7.20 \\ 0.72 \\ 0.07 \\ \hline = 79.99 \end{array}$$

While the conversion of Celsius to Fahrenheit is given as follows:—

$$\frac{9}{5} = 0.180 \times 5.555 = 1 = 0.5555 \times 2 = 1,$$

we have then—

$$32 - \left(\frac{C^{\circ} \times 2}{10}\right) + C^{\circ} \times 2 = F^{\circ}.$$

Example:—

$$32 - \frac{100 C \times 2}{10} = 32 - 20 + 200 = 212^{\circ} F.$$

The advantage which these formulæ present is, as shown by M. Hellmann, that these conversions may be conducted mentally.—I am, &c.,

THOS. PALMER.

4, Rue Haringrode, Antwerp,
November 27, 1897.

ON NEW SPECTRAL LINES OF OXYGEN.

To the Editor of the Chemical News.

SIR,—In the abstracts from foreign journals in the *CHEMICAL NEWS* of November 26 (vol. lxxvi., p. 265), you were good enough to notice my recent discovery of new lines in the spectrum of oxygen, but the wave-lengths, as set forth in the *Comptes Rendus*, have not been given. May I ask you to kindly publish the enclosed copy of a letter from Dr. Schuster, addressed to me in May last, which supplies the omission, and at the same time establishes—by the date—my independent discovery of these lines. The diagrams of the spectrum of oxygen published by Profs. Runge and Paschen in *Wiedemann's Annalen* of July last, and reproduced in the *CHEMICAL NEWS* of November 26th, shows two lines in about the same positions as those which I have observed.—I am, &c.,

H. WILDE.

The Manchester Literary and Philosophical Society,
36, George Street, Manchester, Dec. 7, 1897.

[COPY].

4, Anson Road, Victoria Park,
May 7th, 1897.

DEAR MR. WILDE,

I saw to-day your two new oxygen lines, and asked Mr. Hensalech to measure them so as to make sure they were the same. He finds the wave-lengths 7759 and 7165, agreeing well with yours (7760 and 7160); for with the small spectroscope we had to-day we could not be sure to a few units of these numbers. The lines Mr. Hensalech saw previously must have been nitrogen lines.

Yours sincerely,

(Signed) ARTHUR SCHUSTER.

SUGAR-BEET.

To the Editor of the Chemical News.

SIR,—I shall be glad if you will announce in your next issue that I am distributing sugar-beetroot seed gratuitously to anyone willing to grow sugar-beetroots experi-

mentally next season, and that I will analyse the roots and report thereon free.

The consumption of sugar in the United Kingdom amounts to over one million tons per annum, representing a value of £19,000,000, which all goes to the foreign exporter, and which should be kept in this country if possible.

I was closely connected with the experiments of sugar-beetroot growing carried on in this country about two or three years ago, and by which it was proved that the roots can be grown here quite as well, if not better, than on the Continent, by such well-known landed proprietors as Lord Rosebery, Lord Winchelsea, and Lord Jersey.

Great Britain, with its large consumption of 78 lbs. per head per annum, should grow its own sugar, and thus prevent the closing of any more sugar-refineries, and also be the means of re-opening the large factories now closed in London, Liverpool, Glasgow, Greenock, Bristol, Plymouth, Manchester, and Dublin.

If gentlemen will send me their names and addresses I will forward the seed to them in time for next spring's sowing, and I shall have much pleasure in aiding, gratis, in the formation of any syndicate to erect factories to manufacture the sugar.—Thanking you in anticipation, I am, &c.,

SIGMUND STEIN.

323, Vauxhall Road, Liverpool,
December 1, 1897.

MISCELLANEOUS.

Royal Institution.—The following are the Lecture Arrangements before Easter:—Professor Oliver Lodge, six Christmas Lectures (specially adapted for young people) on "The Principles of the Electric Telegraph"; Professor E. Ray Lankester, eleven lectures on "The Simplest Living Things"; Professor Dewar, three lectures on "The Halogen Group of Elements"; Dr. J. Paul Richter, three lectures on "Some Italian Pictures at the National Gallery"; Professor J. A. Fleming, five lectures on "Recent Researches in Magnetism and Diamagnetism"; Professor Patrick Geddes, three lectures on "Cyprus"; Mr. Wm. H. Hadow, three lectures on "The Structure of Instrumental Music"; Mr. Lionel Cust, two lectures on "Portraits as Historical Documents, Portraits as Monuments." The Friday Evening Meetings will begin on January 21st, when a Discourse will be given by the Right Hon. Sir John Lubbock, Bart., M.P., on "Buds and Stipules"; succeeding Discourses will probably be given by Professor C. Lloyd Morgan, Mr. A. A. Campbell Swinton, Dr. J. Hall Gladstone, Professor L. C. Miall, Captain Abney, Professor J. E. Thorpe, Mr. James Mansergh, the Dean of Canterbury, Professor Dewar, and other gentlemen. Lord Rayleigh will deliver lectures after Easter.

London County Council.—An interesting ceremony took place on Saturday, November 20th, at the Holborn Restaurant, when the members of the staff of the Chemical and Gas Department of the London County Council gave a complimentary dinner to their late chief, Mr. W. J. Dibdin. The new chief of the department, Dr. F. Clowes, of Nottingham College, took the chair, and in proposing the toast of the evening, pointed out that the work carried out by Mr. Dibdin as chief of the department reflected credit alike on the Council and himself. In conclusion, the Chairman presented to Mr. Dibdin, on behalf of the officers of the department, an illuminated address. Mr. Livingston, senior officer of the department, who read the Address to the company, bore testimony to the invariable kindness and courtesy of their late chief, and, referring to his public usefulness, gave as an example his successful treatment of the London sewage. The next

toast, "The Chairman," was proposed by Dr. Teed in an appropriate and somewhat amusing speech. In replying to which Dr. Clowes confided to the company the "nickname" by which he is known among his students at Nottingham, and which promises well for the continuance of the good feeling between chief and officers that has heretofore existed. On account of the recent arduous task imposed upon a sister Department, the toast "The Fire Brigade," proposed by Mr. Livingston, was enthusiastically received. The evening's proceedings were further enlivened by the music provided by Messrs. J. W. Heath and F. Goddard, professionally assisted by Mr. L. Thorne, who artistically rendered "Mary of Argyle," &c., and by Mr. J. W. Kipps, F.R.C.O., A.R.A.M., whose performance at the piano was greatly appreciated. Mr. Edward Minshall favoured the company with two excellent recitations, entitled "The Benediction" and "My First and Last Cricket Match." A vote of thanks to the Hon. Sec., Mr. E. J. Jackman, terminated the proceedings.

Deterioration of Paper.—Sir H. Trueman Wood writes to the *Athenæum*:—A committee of the Society of Arts is now at work on the subject of the deterioration of modern paper. It is a matter of general repute that many books are now printed on paper of so inferior a character that it is liable to perish in a short space of time; but the committee are anxious to have definite examples before them of books that have thus suffered. Might I ask if any of your readers who have had experience of such cases would kindly communicate the facts to me; and also if they would send me any examples of books printed within the last fifty years in which the paper shows signs of perishing? I need not say that any such books will be carefully preserved, and, after the committee have had an opportunity of inspecting them, returned to the lenders.

On the Determination of Silica in Blast Furnace Slag.—G. H. Meeker (*J. Am. Chem. Soc.*).—The dehydration of the silicic acid by means of concentrated sulphuric acid, instead of by heat, is the essential feature of the procedure, for which both rapidity and accuracy are claimed. The filtrate from the silica thus obtained cannot, however, be utilised for the determination of either aluminum or calcium. The method yields satisfactory results in the presence of spinel.

MEETINGS FOR THE WEEK.

MONDAY, 13th.—Society of Arts, 8. (Cantor Lectures). "Gutta Serena," by Eugene F. A. Obach, Ph.D., F.C.S.

WEDNESDAY, 15th.—Society of Arts, 8. "The Purification of Sewage by Bacteria," by Samuel Rideal, D.Sc.

— Microscopical, 8. "A New Form of Photomicrographic Camera and Condensing System," by E. B. Stringer, B.A.

— Institution of Mining and Metallurgy, 8. "Mining on the Black Reef, Witwatersrand Goldfields, South Africa," by W. Fischer Wilkinson. "Notes on Smelting at Broken Hill," by Henry Watson. "Notes on the Buying and Sampling of Ores, and the Working of Mines on the Tribute System, in Chili," by Gerald V. Hopkins.

— Chemical, 8. (Extra Meeting). "Kekulé Memorial Lecture," by F. R. Japp, LL.D., F.R.S.

THURSDAY, 16th.—Chemical, 8. "Stereo-chemistry of Unsaturated Compounds—Part I., Esterification of Substituted Acrylic Acids," by J. J. Sudborough, Ph.D., and Lorenzo Lloyd. "Formation and Hydrolysis of Esters," by J. J. Sudborough, Ph.D., and M. E. Fellmann, B.Sc. "A New Method of Determining Freezing-points in very Dilute Solutions," by M. Wilderman, Ph.D.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Penetration of X Rays.—Could anyone inform me (per *CHEMICAL NEWS*) in what ratio the X rays can penetrate plates of equal thickness of the different metallic elements as regards their atomic weights. Thus, the atomic weight of Fe is 56 and Al 27; seeing that the atomic weight of one is practically double the atomic weight of the other, would Fe offer twice as much resistance to the rays as Al would, and, if not, is there any ratio?—X. Y. Z.

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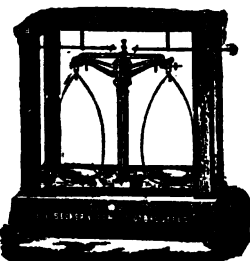


FIG. 11. No. 91.

No. 91. Fig. 11. SHORT BEAM BALANCE for a charge up to 100 grammes in each pan, in french polished glass case, front sliding frame counterpoised, beam divided on both sides in 1-5th part of milligrammes, with new improved arrangement for arrest of pans. Provided with wood stand for taking specific gravity, agate bearings, and sensible to 1-10th part of a milligramme, pans 6 centimetres in diameter, width of bows 9 centimetres. Pans and bows nickel, provided with two rider apparatus, with agate bearings £9 0 0

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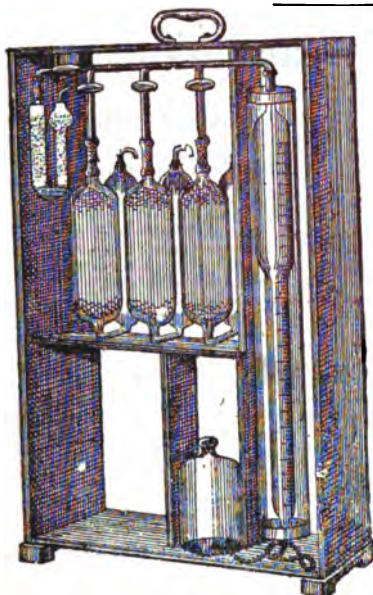
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Edited by
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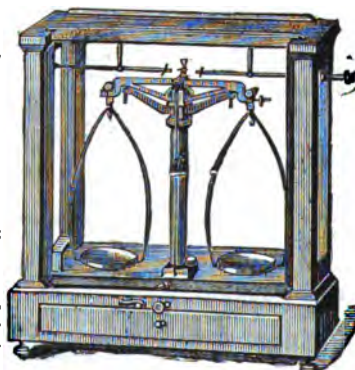


FIG. 3a.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1986.

THE ESTIMATION OF COPPER IN PRESENCE OF OTHER ELEMENTS.

By HARRY BREARLEY.

In a previous paper (CHEMICAL NEWS, vol. lxxvi., p. 189) it was shown that the claim to increased accuracy brought about by the substitution of soda carbonate and soda salts for ammonia and its salts in the cyanometric estimation of copper was a just one. It was also pointed out that the silver iodide end-reaction, which has already been used in the titration of nickel, cobalt, and mercury solutions, could be made supplementary to the colour indicator, or, if need be, act alone.

Two lengthy tables setting forth the influence of certain associated elements on the estimation of copper by cyanide, when performed in the usual way, have already appeared in the CHEMICAL NEWS (Field, i., 61; Thomson, xxxiii., 152), while a writer here and there has chronicled the quantitative behaviour of one or two elements.

It is proposed now to state the observed interference of a similar series of elements, using the AgI turbidity as indicator. The advantage of such an indicator in eliminating the personal error, especially when a precipitate forms in the solution, has been elsewhere commented on.

It must not be presumed that the writer attempts to decide between the differences of previous observers, although the results are not without value in this respect. The aim is to show that in every variety of circumstance the new method of operating is better than the old; the comparisons of Table I. are made to that end.

The new mode of operating is found to be especially serviceable where the associated element is precipitated in the titrated liquid. Very few of such precipitates are entirely without influence on the colour indicator, as Thomson's results show, while others so completely mask it as to make any reliable observations altogether impossible until the precipitate has been allowed to settle. This delay in a process which depends in any degree on the rate at which the reagent is added cannot be satisfactory. Nor can a separation at any stage of the process always overcome this difficulty, on account of the tenacity with which ammoniacal copper solutions adhere to many precipitates, even when the separation chances to be complete.

The observations are summarised in Table I. The following are details common to each test:—Amount of copper, 0.1000 gm.; alkali salts, 20 c.c. HCl (2 normal strength), as chloride; excess of soda carbonate, 30 c.c. (2 N); excess of ammonia, 10 c.c. (2 N). A decreased excess of ammonia was used so as to quite avoid the interchange of silver and copper. Under the head of "Percentage Interferences" are those found in this investigation and those found by Field and Thomson. The soda carbonate series explains itself.

Where the registered interference is below $\frac{1}{2}$ per cent, it may be taken that the element is really without influence, and that such small interferences are due to experimental error. It must be understood that the results stated were obtained by operating in the manner explained in the previous paper. Where the interference is considerable, such attempts as have been made to obviate it, with the measure of success, are stated in the next section.

The behaviour of a method in the hands of different operators is a good index of its value. The ordinary cyanide titration is much better than a comparison of the results of Thomson and Field might suggest. But such

a comparison does show how very susceptible the ammoniacal cyanide procedure is to more or less of what it is desirable should be, in the final operation, inactive reagents.

The Behaviour of the Added Elements. Means of Minimising some Interferences.

The most obvious means of overcoming an interference is to separate the offending element before estimating the copper. Such means need not be further referred to. It is intended here to show how, by modifying the titration, and without any separation whatever in a quantitative sense, the copper may be estimated with sufficient exactness to meet technical demands.

Where the form in which the element was added is not stated, it may be taken that the chloride was used.

Sodium has frequently been shown to be without influence.

Potassium may be presumed to be similarly inactive.

Calcium.—Ammonia, no precipitate. Soda, precipitate easily filterable. The precipitate seems to hold the colour somewhat even when the solution is decolorised. A greater excess of cyanide is therefore advisable. Thomson explains his high result by presuming that there is some reaction between the cyanide and the calcium salts. His cyanide contained considerable quantities of carbonate. May not the adherent colour more truly account for the error?

Barium and Strontium.—Similar remarks apply to these metals.

Magnesium.—No precipitate in either case.

Zinc.—More than usual interest attaches to this element, because the alloys of copper and zinc are so abundant. On this account the greatest pains have been taken to minimise its influence. Under this head will be noticed a number of modifications which may be applied to other elements.

Zinc-Ammonia.—An excess of 10 c.c. 2 normal ammonia is not sufficient to precipitate the zinc and re-dissolve it; 30 c.c. was used. The most noticeable feature of titration is the large excess of cyanide, over and above that actually required to combine with the zinc, needed to discharge the colour.

Zinc-Soda.—On making alkaline with soda carbonate the zinc is of course precipitated. On adding the cyanide the colour goes as usual, until the theoretical amount of cyanide is added. It is possible at this point to get a colourless solution, by prolonged standing, holding the greater part of the zinc precipitated; but the precipitate is not colourless. It remains a pale green in the most persistent manner until enough cyanide has been added to dissolve it completely, or nearly so. The excess of cyanide required to do this when copper and zinc were together in equal amounts varied from 60 to 80 per cent more—according to volume, time of standing, &c.—than was needed to react with the copper alone.

On adding the potassium iodide, and then the silver nitrate, there appeared a white precipitate, in many ways unlike the creamy silver iodide. It was filtered off, more AgNO₃ added, and again the white precipitate appeared, and so on the precipitation and filtration might alternate until the silver iodide turbidity did appear.

This at once suggests that the zinc which had combined with the cyanide is displaced by the silver, and that the repeated precipitations and filtrations are unnecessary if enough silver nitrate is at once added to replace the whole of the zinc.

In doing this it was possible to use the potassium iodide to indicate when enough silver had been added. It is not difficult to observe this point when as much as 0.1 gm. of zinc is present, because the zinc carbonate, previously translucent and white, becomes, owing to admixed AgI, opaque and yellowish, and, instead of settling in flakes, remains suspended in no determinate shape. But the indication is hardly delicate enough to accept as the end of the operation, and so a little cyanide is added, which

TABLE I.

METAL.	AMMONIA SERIES.						SODA CARBONATE SERIES.				
	Copper found.			Percentage error.			Copper found.			Percentage error.	
	0.01	0.05	0.10	H. B.	Thomson.	Field.	0.01	0.05	0.10		
Grm. added	0.01	0.05	0.10	H. B.	Thomson.	Field.	0.01	0.05	0.10		
Calcium	0.1000	0.1000	0.1005	0.5	1.35	0.0	0.1000	0.0997	0.1000	0.0	
Barium	—	0.1000	0.0996	0.4	1.90	0.0	0.1000	0.1002	0.1000	0.0	
Strontium	—	0.0998	0.1000	0.0	2.55	0.0	—	0.1002	0.1000	0.0	
Magnesium	—	0.1004	0.1002	0.2	0.90	0.0	—	0.0998	0.1002	0.2	
Zinc	1012	0.1054	0.1094	9.4	20.65	25.0	—	0.1015	0.1020	2.0	
						34.6*					
Cadmium	—	0.1010	0.1028	2.8	6.85	—	—	0.1004	0.1000	0.0	
						15.4*					
Aluminium	0.1003	0.1006	0.1007	0.7	3.55	0.0	0.1006	0.1007	0.1007	0.7	
Iron	0.1002	0.1002	0.0998	0.2	0.85	0.0	0.1000	0.0994	0.0987	1.3	
Manganese	0.0968	0.0964	0.0969	3.1	24.85	12.0	0.0977	0.0966	0.0970	3.0	
Chromic oxide	—	0.0869	0.0730	27.0	—	0.0	0.0992	—	0.0975	2.5	
Chromic acid	—	—	—	—	—	—	—	0.1002	0.0996	0.4	
Tin	—	0.1000	0.0998	0.2	1.35	0.0	—	0.1000	0.1003	0.3	
Molybdenum	—	0.0998	0.1000	0.0	0.15	—	—	0.1004	0.1002	0.2	
Arsenic	—	0.0998	0.0998	0.2	0.0	0.0	—	0.0998	0.0998	0.2	
Antimony	—	0.1000	0.1004	0.4	1.9	0.0	—	0.1002	0.1004	0.4	
Bismuth	—	—	—	—	2.45	0.0	—	0.1002	0.1000	0.0	
Lead	—	0.1002	0.1000	0.0	0.4	0.0	—	0.0998	0.1002	0.2	
Mercury (ous)	—	0.1062	0.1120	12.0	19.1	—	—	1070	113.9	13.9	
" (ic)	—	0.1089	1182	18.2	23.2	50.0	0.1018	0.1107	0.1200	20.0	
Uranium	—	0.1004	0.1004	0.4	2.45	—	—	0.0998	0.1002	0.2	

* Dulin, *Journ. Amer. Chem. Soc.*, May, 1895.

dissolves the AgI without dissolving more than traces of the zinc. The solution is then filtered and finished in the ordinary way. An alternative is to add an excess of silver nitrate, but no potassium iodide, filter, and titrate the excess with cyanide. The soda results in Table I. were obtained in this way.

Such a procedure as the above even apart from the error cannot be widely serviceable, on account of the expense. An attempt was made to substitute nickel for silver, for economy's sake, and with the idea that the error on using silver was possibly due to its inability to completely displace the zinc. The fear was that nickel, being more active to cyanide than silver, might also displace the copper. This, however, it did not do, but being only partially soluble in soda carbonate it did necessitate an examination of the residue, as well as the filtrate, before the excess could be found. The multiplicity of operations pending such a procedure led to its rejection.

One point, which ought never to have been overlooked, was strongly emphasised; that is, the nickel may be associated with the zinc as carbonate in a solution which contains an excess of free cyanide. This recalls the tenacity with which the copper was retained in the zinc carbonate, and suggests that an examination of the residue might have revealed the amount of silver necessary to correct the 2 per cent error.

Other reagents, such as tartaric and citric acids, were tried. They did not eliminate the error. They are objectionable, too, for reasons stated below.

The most satisfactory modification lies in the use of sodium pyrophosphate (prepared by heating Na_2HPO_4 to redness). A saturated solution was used. Its influence on the titration of pure copper solutions is shown below:—

0.0	25.0	50 c.c. pyrophosphate.
0.0500	0.0504	0.0502 gr. Cu indicated.

This reagent was suggested by T. Moore (*CHEM. NEWS*, lix., 160), for titrating nickel in presence of zinc. Its only defect is a slight lagging in the reaction between the cyanide and silver iodide which accompanies its use.

This lagging may be counterbalanced by adding the same volume of pyrophosphate to the standard copper. Two results obtained in this way are:—

0.0	0.05	0.1 gr. zinc present.
0.1000	0.1008	0.1006 gr. copper found.

In other hands it has given as good and better results.

Cadmium.—The reactions very much resemble zinc, but of much less intensity. The precipitate formed on making alkaline is in each case dissolved by the cyanide. With sodium carbonate the cadmium was re-precipitated on adding the silver nitrate, as explained above, and as no great excess of cyanide is needed, and the titration offers no other difficulty, there was no need to try further modifications, although doubtless the pyrophosphate would answer with cadmium as well as with zinc.

Aluminium.—There is a precipitate with either alkali. These precipitates are objectionable in that they are difficult to filter off, and the prolonged exposure of even dilute cyanide solutions tends to give high results.

The difficulty, and a similar one with other elements, may be overcome in several ways:—

I. Fractional filtration; but great care must be taken that the fraction preserves its proportionate volume throughout the succeeding operations. It is a good plan to fractionate the standard similarly.

II. The bulk of silver nitrate necessary to combine with the free cyanide may be added before filtration. This precaution may also be taken along with fractional filtration. It minimises the effect of prolonged exposure, and may be safely used for any of the elements (except chromium) in Table I., without fear of the objectionable features accompanying zinc carbonate.

III. The formation of precipitate may be prevented. This can be done by adding tartaric or citric acid, soda pyrophosphate, or making alkaline with soda or potassium hydrate. The two acids so generally employed for similar purposes cannot be recommended here. So small a quantity, about 1 grm. of tartaric acid (say), as suffices to keep 0.1 grm. of aluminium in solution has a very objectionable influence on the titration. The colour of the alkaline solution is less deep than without the acid, and as the proportion of aluminium rises the later colourations on adding cyanide shade off into pale green, and are of little use as indicators; but the most objectionable feature is the way in which a turbidity once formed gradually disappears, so that one is left at the mercy of all manner of evil suggestions, however conscientiously the work may

be done. It may be possible to wait for a disappearing turbidity, and replace, and so on, until all errors vanished, but such a procedure is not practicable. It is this defect, in a much smaller degree, which was referred to under zinc as accompanying pyrophosphate. Citric acid behaves similarly. These remarks are based on an observance of soda solutions only.

Seven or eight of the tabulated elements give no precipitate when soda carbonate is replaced by soda hydrate. The behaviour of such solutions may be mentioned, but the procedure can be recommended only within narrow limits. There should not be a greater excess than 10 c.c. 2 normal NaHO in about 250 c.c. of solution, else the displacement of Cu by silver takes place; and the standard should be worked alongside the sample, because the turbidity after standing awhile becomes brown.

(To be continued).

ZINC IN WATER.

By PERCY A. E. RICHARDS, F.I.C.

As the contamination of water by zinc is not of very frequent occurrence, perhaps the following instance may be of interest:—

The water was drawn from a Berkshire district, and, after being stored in a reservoir, was supplied to a private residence for drinking purposes, by a galvanised iron pipe about 2 miles in length.

When first received the sample was perfectly bright and clear, but when left exposed to the air for about an hour it developed a distinct scum on the surface.

The following results were obtained from an analysis of the water:—

Free ammonia	0.0042 grain per gallon.
Albumenoid ammonia..	0.0028 " "
Nitrogen as nitrates ..	0.035 " "
Chlorine as chlorides ..	1.3 grains "
Total solid constituents	14.35 " "
Zinc bicarbonate	5.12 " "
Iron	traces.

The presence of the zinc was easily detected in the unconcentrated water by both the ammonium sulphide and potassium ferrocyanide tests. Upon boiling the water a precipitation of carbonate of zinc took place.

The "total hardness" of the water drawn direct from the reservoir was equivalent to 4.5 grains of calcium carbonate, and the total solid constituents were only 10 grs. per gallon.

ESTIMATION OF CHLORINE, BROMINE, AND IODINE IN SALINE WATERS.

By PERCY A. E. RICHARDS, F.I.C.

VARIOUS methods have been suggested at different times for the separation of the halogens from mixtures of the three. For instance, Donath has propounded a process based on distillation with chromic acid, as a means for the separation of bromine.

Again, Vortmann recommends boiling a known volume of the solution containing the three halogens, in the first place with pure manganese dioxide and acetic acid. This causes the liberation of the whole of the iodine, and this can then be boiled off. The chloride and bromide in the residue may next be estimated in terms of decinormal silver nitrate solution, when the difference between this number and the equivalent in silver nitrate for the same volume of the untreated water gives the amount of decinormal iodine present.

A known volume of the water is next boiled with per-

oxide of lead and acetic acid, and the bromine and iodine thus liberated boiled off. The silver nitrate equivalent of the residue is determined as before, and the bromine calculated by difference between this number and the previous determination.

These processes give satisfactory results so long as one of the halogens is not present in much greater amount than either of the other two. But in the examination of waters it is almost invariably the case that the amount of chlorine greatly exceeds that of both bromine and iodine, sometimes to the extent of 1000 or 2000 to 1. In cases like these, the estimation of iodine and bromine by difference is of very little value. I had occasion recently to examine some waters of this character, and found that the following methods gave very good and concordant results:—

In the first place, the total halogen equivalent in terms of decinormal silver nitrate solution was accurately determined. The iodine present was then estimated by treating 250 or 500 c.c. with acetic acid and hydrogen peroxide for about half an hour, and extracting with chloroform. The mixture was shaken in a separating funnel, with successive small quantities of chloroform, and these washings added to the first portion. The solvent was then well washed with distilled water to remove any peroxide of hydrogen, and was then titrated with decinormal sodium thiosulphate solution, and the amount of iodine calculated. This process, which is due to Cook, is simple and accurate.

The bromine was next estimated by shaking the iodine-free liquid left in the separating funnel, with chlorine water and chloroform, avoiding much excess of the former. The solvent was drawn off, and the aqueous solution shaken several times with small quantities of chloroform, the latter being added to the first portion. Any chlorine present is washed out of the chloroform by repeated agitation with distilled water, and the bromine estimated by adding a few crystals of iodide of potassium and titrating with thiosulphate of soda solution.

The iodine and bromine present being now known, the silver nitrate equivalents can be calculated and subtracted from the total halogen equivalent, and from this result the chlorine present is deduced.

This process enables the bromine and iodine to be estimated directly and with considerable accuracy, and is therefore specially suited to the examination of saline waters where the quantities present are relatively small.

A REVISION OF THE ATOMIC WEIGHT OF NICKEL.*

FIRST PAPER.—THE ANALYSIS OF NICKELOUS BROMIDE.

By THEODORE WILLIAM RICHARDS
and
ALLERTON SEWARD CUSHMAN.

(Continued from p. 285).

Purification of Materials.

Nickel.—Since the possibility of preparing and analysing pure nickelous bromide had now been proved, the next step was to purify all the materials concerned in its manufacture. First among these materials comes nickel, which must not only be freed from all known impurities, but must also be so treated as to detect and eliminate unknown ones. With this latter purpose in view, our material was obtained from two distinct sources; first, the "pure" nickel of commerce; and, secondly, really pure nickel (containing only a little iron) prepared by Dr. Mond

* Contribution from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, Vol. XXIII., No. 7.

through the carbonic oxide process and kindly presented by Dr. Wolcott Gibbs.

It is convenient to consider first our treatment of the commercial nickel. In proceeding with the further purification of this sample, our first step was to remove the metals of the copper and tin groups. The simple treatment with hydric sulphide has generally been considered sufficient to insure the separation of the metals of these groups, in spite of the fact that many of the sulphides when present in small quantities often assume a colloidal condition in which they cannot be separated by filtration. In our case this difficulty was avoided by regulating the acidity of the solution so that a certain amount of black nickelous sulphide was precipitated, which effectually "swept" this liquid, coagulating small quantities of foreign sulphides. After filtration the liquid was boiled to drive off the hydric sulphide, oxidised with a few drops of nitric acid, made alkaline with ammonia, and filtered. The precipitation of the sulphide was now continued by the passage of a little washed hydric sulphide. This first comparatively small amount of sulphide was filtered out and discarded. The remaining nickel was then as nearly as possible completely precipitated in the form of sulphide. After the precipitate had been allowed to settle in a closed flask over night, the liquid was decanted, and the precipitate was washed by decantation many times with boiling water until neutral. Cold dilute hydrochloric acid was now added, and the precipitate was digested for several days. Since the colour of the supernatant liquid showed that a slight amount of even the comparatively insoluble nickel sulphide had gone into solution, the assumption was not unreasonable that nearly, if not quite, all of the more soluble sulphides must have been removed. The black precipitate was now repeatedly washed with hot water until the washings were quite neutral; it was next dissolved in strong hot hydrochloric acid, and, after the separated sulphur had been removed, the solution was evaporated to dryness, and the residue was taken up with water. The material was now considered fairly free from its usual impurities, with the single exception of cobalt.

In commenting on the work of an early experimenter upon the atomic weights of nickel and cobalt, Clarke has objected that "his results are entitled to no especial weight at present, since it cannot be certain from any evidence recorded that the oxide of either metal was absolutely free from traces of the other" ("Re-calculation," 1897, p. 291). Since the two metals have atomic weights only differing at the outside by half a unit, "traces" of one in any preparation of the other metal could not alone furnish a reason for invalidating the results. Nevertheless, for our purpose it seemed desirable to prepare nickel as nearly free from cobalt as possible. In order to attain this end with any degree of certainty, it is obvious that a qualitative test must be found that should show with sufficient accuracy the presence or absence of cobalt. Winkler (*Zeit. Anal. Chem.*, vi., 20) has recommended a test for which he claimed greater accuracy than the better known method with potassium nitrite. The moderately dilute solution of nickel is treated with ammonia until a clear blue colour is obtained, and then one or two drops of potassic permanganate are added. If no cobalt is present, the blue solution of nickel takes on a purple tinge; whereas cobalt, if present, reduces the permanganate. Winkler does not state the dilution of the permanganate solution, or how much should be added, although manifestly the degree of refinement of the test depends on these points. The permanganate solution which worked well with us contained 1 grm. of the salt in a litre. To the dilute solution of nickel to be tested, contained in a colour-comparison apparatus, enough ammonia is added to render the solution a light sky-blue, and then one-tenth of a c.c. of the permanganate is dropped in. Under these conditions, the mixture appears decidedly purplish in hue, if cobalt is absent. Of course, the test is of value only in the absence of any foreign substances having either

a reducing or an oxidising action on permanganate. We have found it possible by this method to detect one part of cobalt in 2500 parts of nickel, an amount of impurity which could cause a final error in the atomic weight of only 1 part in 500,000.

Anthon's process (see Dammer, *Anorg. Chem.*, iii., 490) for eliminating cobalt was adopted for the purification of our sample. The nickel was twice fractionally precipitated as hydroxide by means of pure sodic hydroxide, the mixture being thoroughly boiled. As far as our test could show it, all the cobalt remained in the filtrate, the last precipitate being contaminated only with a small amount of alkali.

When ammonia is added to a solution of nickelous bromide, a beautiful violet crystalline compound is formed, having the formula $\text{NiBr}_2 \cdot 6\text{NH}_3$, according to our analysis as well as those of Rammelsberg (*Pogg. Ann.*, lv., 243). Since this compound is characteristic of nickel, and similar compounds are not formed by cobalt or most other metals under similar circumstances, and since it is soluble in strong hot ammonia water, but almost insoluble in cold ammonia, it affords a very convenient and effectual means of purifying nickel preparations. Our purified oxide was hence dissolved in pure hydrobromic acid, ammonia was added in excess, and the mixture contained in a platinum flask, was cooled to zero. The beautiful purple precipitate was collected upon pure filter-paper and was washed with strong ammonia. All the material used in the analyses was passed through this treatment at least once, although the various samples were subsequently subjected to different methods of further treatment which will be described in each case.

The violet compound made from our first sample of purified nickel was treated with an excess of water and boiled in a platinum dish, a proceeding which completely precipitated the nickel as hydroxide. The greenish mass was thoroughly washed, and was then dissolved in hydrobromic acid. The nickelous bromide thus obtained was dried in a vacuum desiccator over dry soda; but even after this treatment it was found to have retained considerable quantities of water, an impurity which greatly increased the difficulties of sublimation. Hence this sample, numbered I., was used only for two preliminary analyses.

Another portion of this same sample of the violet compound was re-crystallised several times in succession by cooling its hot ammoniacal solution. The resulting magnificent crystals were dissolved in water and the solution was boiled to drive off the ammonia. The precipitation being thus accomplished, the basic hydroxide was collected, carefully washed, dried, and ignited over an alcohol flame in a porcelain vessel. The resulting nickel oxide was reduced to the metal by igniting this material, held by a porcelain boat in a combustion-tube through which a current of pure dry ammonia gas was passing. The spongy nickel thus produced was changed to bromide and sublimed in the manner already described. The pure substance thus prepared is designated below as Number II.; it also served for two of the earlier analyses.

The further purification of the sample of nickel obtained by the process of Mond, Langer, and Quincke, at first proceeded exactly in the steps just described; except that no attempt was made to remove cobalt, since none was present. After it had reached the stage of treatment represented by the last hydroxide obtained above, the material was converted into the sulphate and subjected to electrolysis out of an ammoniacal solution in a platinum dish. The object of this procedure was, of course, to free the nickel still more effectually from the alkalis, silicas, and many other impurities which are not precipitated on the cathode. The bright heavy deposit of pure nickel was dissolved, with a great deal of difficulty, in re-distilled strong nitric acid; and the excess of acid was driven off by evaporation. Ammonia and a large excess of pure water were now added, and the solution was boiled until the basic hydroxide was completely precipitated. This

was subsequently changed to metallic nickel and then into the sublimed bromide, in the manner already described, and the resulting material, labelled III., served for a large number of analyses. A small portion of it, that used for Experiment 8, was re-sublimed.

The sample of material used for the final analysis was even more carefully purified than this, however. A quantity of the pure nickelous oxide of about the grade of purity of Sample III., coming originally from the Mond nickel, was dissolved in sulphuric acid, and the solution was made alkaline by passing in pure ammonia gas, in a platinum dish. When most of the nickel had been deposited electrolytically from this solution, the portion remaining in the electrolyte was thrown away. The bright coating of nickel was washed, pure dilute sulphuric acid

Thus, while all of our samples of nickelous bromide analysed had been sublimed, the several samples had received previous to sublimation very varying treatment. The fourth had been put through a process of purification far more searching than the first, which had merely been freed from the ordinary known impurities. Hence the essential identity of the results obtained from these several samples is very striking.

Purification of other Materials.—Silver was purified exactly in the manner described by Richards and Parker (*Proc. Amer. Acad.*, xxxii., 62) in a recent paper upon the atomic weight of magnesium. The electrolytic crystals were finally fused upon a boat of pure lime in a vacuum. For further details the above-mentioned paper should be consulted.

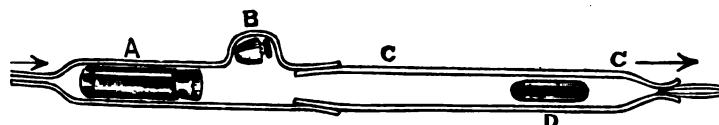


FIG. 2.—BOTTLING APPARATUS, HORIZONTAL SECTION.

A, Weighing bottle. B, Stopper of bottle. C C, Hard glass tube. D, Platinum boat containing nickelous bromide.

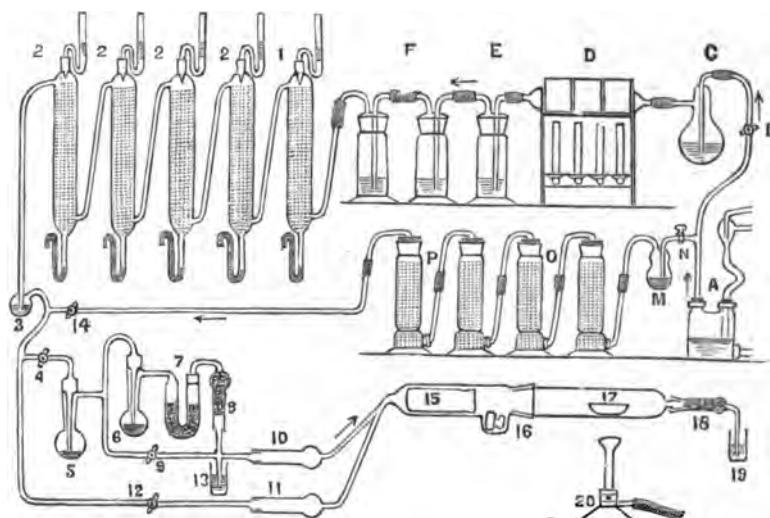


FIG. 3.—APPARATUS FOR IGNITING NICKELOUS BROMIDE IN ANY DESIRED MIXTURE OF GASES.

The use of rubber was confined to the first part of this train, where it could do no harm (A B C D E F and A M N O P).

was put into the dish, and with reversed poles a strong current was sent through the solution until nearly, but not quite, all of the nickel was dissolved. The solution was then decanted into another dish, ammonia was passed in until the precipitate formed had re-dissolved, the poles were again reversed, and then nickel was once more almost all deposited. This cycle of operations, which gives an excellent method of fractionation, was repeated three times. The final deposit of nickel was dissolved by filling the platinum dish with pure dilute nitric acid and reversing the poles. Only one who has tried dissolving a deposit of nickel on a platinum dish, even in strong nitric acid, can appreciate the ease, cleanliness, and convenience of this method of procedure. The solution of nickel nitrate thus prepared was concentrated by evaporation, and ammonia was passed in until a mass of crystals of the blue ammonio-nickel nitrate was formed. After the mother liquor had been poured off, the crystals were washed with pure ammonia water, and were finally boiled in an excess of pure water in the same platinum dish. The resulting basic hydroxide was then changed to spongy nickel and nickelous bromide in the usual fashion, bearing the title No. IV.

With the co-operation of Mr. Baxter, bromine was purified in a preliminary fashion by solution in strong aqueous calcic bromide, and a subsequent separation. Carefully washed red phosphorus was used to convert the bromine thus obtained, after it had been several times re-distilled, into hydrobromic acid, and the hydrobromic acid was freed from iodine and organic matter by several fractional distillations with bromine water. From this pure hydrobromic acid, bromine was obtained by means of manganese dioxide free from chlorine. 2.10289 grms. (in vacuum) of silver yielded 3.66066 grms. (in vacuum) of argentic bromide on combination with this bromine,—a ratio of 57.445 : 100.00. Mr. Baxter found 57.444 in a similar experiment, while Stas's value was 57.445 : hence the purity of our bromine and silver was proved.

Sodic hydroxide was freed from most metallic impurities (iron, &c.) by electrolysis. Ammonia was re-distilled in platinum vessels, as were also nitric and hydrochloric acids. Sulphuric acid was distilled in glass, alkalis being a less dangerous impurity than platinum in the instances where it was used. Water was purified by distillation,

first from alkaline permanganate solution, and then with a trace of acid potassic sulphate.

In processes where the presence of bromine rendered the use of platinum impossible, Jena glass, or at high temperatures Berlin porcelain, was used. For some of the platinum and other apparatus we are indebted to the Cyrus M. Warren Fund for chemical research in Harvard University.

The Method of Analysis.

Turning now to the method of analysing the carefully prepared nickelous bromide, it is obvious that the first point to be considered is the accurate determination of the weight of the salt to be analysed. This process was effected by means of apparatus similar to that devised for the drying and weighing of magnesic chloride, and described in a recent paper by Mr. H. G. Parker and one of us (*Proc. Amer. Acad.*, xxii., 58), upon the atomic weight of magnesium. In this apparatus, constructed wholly of glass by Mr. Baxter, the bromide under consideration, contained in a platinum boat, was ignited at about 400° in a stream of mixed nitrogen and hydrobromic acid until constant in weight. It was then allowed to cool in a stream of pure dry nitrogen, and when cool it was pushed in pure dry air into its weighing bottle, which was immediately closed by a mechanical device. In this fashion it is possible to dry and weigh accurately the most hygroscopic of substances, and repeated ignitions of the same specimen have shown that perfect constancy in weight may thus be obtained. It is hard to believe that any water is retained by nickelous bromide at 400°, and certainly none could be absorbed during the cooling, for the whole apparatus was shut off from the outside air, and all the gases admitted were first passed through phosphoric oxide.*

The bromide in question was then weighed by substitution, using as the tare to be substituted a weighing bottle precisely like the one containing the platinum boat and substance. In this way alone can the weight of a large bottle be determined within the fraction of a tenth of a m.grm. The balance has already been described in detail (*Proc. Amer. Acad.*, xxvi., 242); the weights were of course compared and standardised with great care, and were used for no other work during the progress of this.

Having been weighed with accuracy, the nickelous bromide was dissolved in pure warm water in a flask, and from this was transferred to the large beaker flask in which the precipitation was to take place. The platinum boat in which the salt had been treated remained invariable in weight, showing that it had not been attacked by hydrobromic acid at a high temperature.

As has been said already, the salt used in the preliminary series was contaminated with a small amount of nickelous oxide, which was filtered off and weighed. The amount of this impurity is given simply to show that the slight irregularity of the results was not dependent upon the adulteration; the weights of nickelous bromide given are those left after the subtraction of the weight of the oxide. All the bromine contained in the solution was precipitated in these seven analyses by means of an excess of argentic nitrate, and the argentic bromide was collected and weighed with the usual precautions.

(To be continued).

Estimation of Small Quantities of Methyl-alcohol, Formic Aldehyd, and Formic Acid.—M. Nicloux.—The author has extended his method for the estimation of alcohol by the reduction of bichromate of potash in the presence of sulphuric acid, to the estimation of methyl alcohol, formic aldehyd, and formic acid.—*Bull. Soc. Chim. de Paris*, Series 3, xvii.-xviii., No. 16-17.

* A detailed description of this apparatus will be given in a future paper, upon Cobalt.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, November 18th, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

MESSRS. W. J. Elliott, H. S. Elworthy, F. F. de Morgan, Frank Moul, A. Harden, and Charles E. Brown were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Cecil Joslin Brooks, 24, Wood Street, Woolwich; Charles Henry Burge, Iddesleigh Crescent Road, Kingston Hill, S.W.; M. J. Cannon, 101, The Chase, Clapham Common, S.W.; William Ransom Cooper, M.A., B.Sc., 87, Upper Tulse Hill, S.W.; Frederick Robertson Dodd, 1, Wesley Street, Liverpool; Jules Fuerst, 23, Marlborough Road, N.W.; James Brown Reid, 6, Southfield Terrace, Skepton; Harold Charles Sayer, Devon Villa, Summerhill Road, Dartford.

The PRESIDENT announced that he had received a letter from Sir Fleetwood Edwards stating that he had been commanded by the Queen to forward to him a Medal in commemoration of the 60th Anniversary of Her Majesty's reign.

Of the following papers those marked * were read :—

*118. "On the Decomposition of Camphoric Acid by Fusion with Potash or Soda." By ARTHUR W. CROSSLEY and W. H. PERKIN, jun.

The authors find that by the action of fused potash on camphoric acid a very complicated mixture of acids is obtained. The volatile portion consists of acetic, propionic, isobutyric, isovaleric, and methylisopropylacetic acids, together with acids of the formulæ $C_6H_{11}CO_2H$, $C_7H_{13}CO_2H$, and $C_8H_{17}CO_2H$, of which the constitution is doubtful. The non-volatile acids consist of pimelic (isopropylsuccinic) acid and a new substance, dihydrocamphoric acid.

Dihydrocamphoric acid, $C_{10}H_{18}O_4$, crystallises in nodular masses melting at 105–106°, and on oxidation with dilute nitric acid yields succinic acid, oxalic acid, and an acid of the formula $C_8H_{14}O_4$, which is in all probability the $\alpha\beta$ -trimethylglutaric acid described by Balbiano.

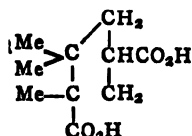
On treatment with acetic anhydride, dihydrocamphoric acid is converted into its anhydride, which, when heated to boiling, decomposes with elimination of carbon dioxide and formation of a cyclic ketone of the formula $C_{10}H_{16}O$. *Dihydrocamphorone* is a liquid boiling at 180–181° and smelling strongly of peppermint. It forms a liquid ketoxime and a semicarbazone melting at 202–203°.

The results obtained on fusing camphoric acid with caustic soda differ markedly from the above. The lower volatile fatty acids appear to be the same as those obtained from caustic potash, but there is also present an unsaturated acid of the formula $C_8H_{12}CO_2H$.

The acids not volatile in steam consist of large quantities of pimelic acid and some unchanged camphoric acid, together with two new acids, pseudocamphoric acid, $C_{10}H_{16}O_4$, and an acid of the formula $C_9H_{16}O_4$, boiling at 254–257° at 50 m.m.

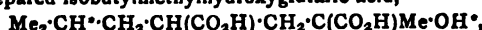
Pseudocamphoric acid crystallises from water in colourless six-sided plates with bevelled edges, usually grouped together in the form of stars, and melts at 119–120°. It forms a crystalline anhydride melting at 52–53° and an anilic acid melting at 208°. A further difference from its isomeric *d*-camphoric acid is that when treated with sulphuric acid it does not evolve carbon monoxide forming a sulphonic acid.

The authors explain the results of the action of fused alkalis on camphoric acid and deduce constitutional formulæ for the various compounds obtained on the assumption that camphoric acid is represented by the formula—

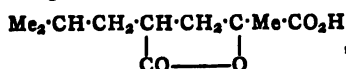


*119. "Experiments on the Synthesis of Camphoric Acid." By W. H. BENTLEY and W. H. PERKIN, jun.

The authors have attempted to prepare an acid of the constitution suggested (*Proc.*, 1896, xii., 189) as a probable formula for camphoric acid (see preceding abstract). They prepared isobutylmethylhydroxyglutaric acid,—



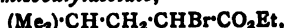
but did not succeed in eliminating water in the direction desired (at the points *), the product in most of the experiments being the lactone-acid—



or one of its derivatives.

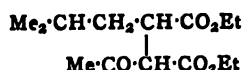
The following substances were prepared during the course of the work.

Ethyl bromoisobutylacetate,—

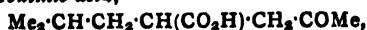


an oil boiling at 100—103° (17 m.m.).

Ethyl acetylisobutylsuccinate,—

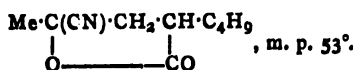


prepared by the action of ethyl bromoisobutylacetate on ethyl sodioacetate. It is a colourless oil boiling at 160° (25 m.m.), and when hydrolysed with dilute hydrochloric acid or sulphuric acid yields isobutylsuccinic acid, $\text{Me}_2\text{CH}\cdot\text{CH}_2\cdot\text{CH}(\text{CO}_2\text{H})\cdot\text{CH}_2\cdot\text{CO}_2\text{H}$, m. p. 109°. When hydrolysed with concentrated hydrochloric acid, however, isobutyllevulinic acid,—

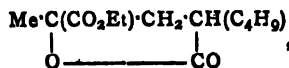


is formed. This is a colourless oil boiling at 190° (30 m.m.), and yielding a semi-carbazone melting at 192: bromine, in the presence of potash, oxidises it to isobutylsuccinic acid.

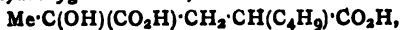
Isobutyllevulinic acid readily unites with hydrogen cyanide, forming isobutylhydroxycyanovaleic acid, $\text{Me}\cdot\text{C}(\text{OH})(\text{CN})\cdot\text{CH}_2\cdot\text{CH}(\text{C}_4\text{H}_9)\cdot\text{CO}_2\text{H}$, which crystallises with 1H₂O in needles melting at 95—96°. This acid on distillation yields the corresponding lactone,—



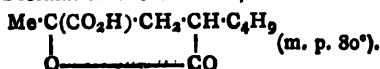
When an alcoholic solution of the hydroxy-cyano acid is saturated with hydrogen chloride, it is hydrolysed and converted into the ethereal salt of isobutylmethylhydroxyglutaric acid, $\text{Me}\cdot\text{C}(\text{OH})(\text{CO}_2\text{Et})\cdot\text{CH}_2\cdot\text{CH}(\text{C}_4\text{H}_9)\cdot\text{CO}_2\text{Et}$. On distillation, this loses alcohol, forming the ethereal salt of the lactone of isobutylmethylhydroxyglutaric acid,—



which is an oil boiling at 168° (17 m.m.). This on hydrolysis with alcoholic potash is converted into isobutylmethylhydroxyglutaric acid,—



a crystalline acid melting at 134° with elimination of water and formation of the lactone,—

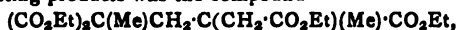


*120. "Synthesis of an Isomeride of Camphoric Acid." By S. B. SCHRYVER, Ph.D.

The compound—



was synthesised by acting on methylacrylic ester, $\text{CH}_2\text{C}(\text{Me})\text{CO}_2\text{Et}$, with sodiummethylmalonic ester. The addition product thus obtained, having the formula $(\text{CO}_2\text{Et})_2\text{C}(\text{Me})\cdot\text{CH}_2\cdot\text{CNa}(\text{Me})\text{CO}_2\text{Et}$, instead of being isolated, was acted on by iodicacetic ester. Among the resulting products was the compound—

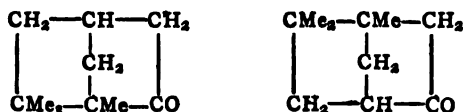


which on hydrolysis gave an acid of the required formula. This was isolated by means of its lead salt, which is insoluble in acetic acid, in the form of a syrup, and this on treatment with nitric acid gave a crystalline oxy-acid having the formula $\text{C}_9\text{H}_{14}\text{O}_7$. It is, therefore, an isomeride of camphoric acid.

DISCUSSION.

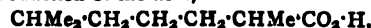
Dr. KIPPING said that it was very difficult to criticise papers which, like those read by Dr. Crossley, contained so many new and important facts relating to substances of somewhat complex constitution. It seemed to him, however, that all the results obtained by Dr. Crossley and Dr. Perkin in their study of the substances produced by fusing camphoric acid with potash, could be explained on the basis of Bredt's formula; such an opinion might of course be altered after carefully examining the details of the work as laid out in the published papers.

Dr. FORSTER pointed out that the formula for camphoric acid employed by Drs. Crossley and Perkin appears to involve the expression of the constitution of camphor by one of the formulae—



The behaviour of camphoroxime on dehydration, and the isomerism of two series of campholenic derivatives, seem to render the second expression the more probable of the two, but before either could be accepted, an explanation must be furnished of the changes involved in the production of such compounds as cymene and carvacrol from camphor—changes which, it must be remembered, are readily explained by Bredt's formula.

The production of the acid,—



obtained by the authors by fusing camphoric acid with alkali, harmonises with the new camphoric acid formula, but it must not be overlooked that Bredt's formula for camphoric acid affords an equally plausible explanation.

Dr. CROSSLEY, in reply, said that the main evidence for accepting the formula proposed by Professor Perkin was embodied in his paper on sulphocamphylic acid, which had not yet been published in detail. Whilst the production of most of the compounds described by Professor Perkin and himself admit of explanation by Bredt's formula, it does not account for the formation of $\alpha\beta\beta$ -tetramethyladipic (dihydrocamphoric) acid.

*121. "The Action of Magnesium on Cupric Sulphate Solution." By FRANK CLOWES, D.Sc., and R. M. CAVEN, B.Sc.

The authors have examined the action of magnesium on solutions of cupric sulphate of different strengths, both at atmospheric temperature and at a temperature near their boiling-point. They find that the evolution of hydrogen which always takes place is accompanied by the precipitation of a mixture of cuprous oxide and metallic copper in proportions which vary with the conditions of the experiment.

When a dilute solution of cupric sulphate is employed, the above-mentioned products are accompanied by a

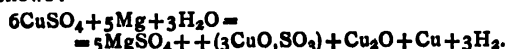
quantity of a green substance, which consists of a mixture of basic hydrated sulphates of copper and magnesium. This substance was observed to form when a saturated solution of cupric sulphate was employed, but it was decomposed again before the reaction was completed.

The time of the reaction varies from ten minutes in the case of a hot strong solution of cupric sulphate to several days, or even a week, when a dilute solution is employed at atmospheric temperature.

The quantities of the three reduction products, cuprous oxide, copper, and hydrogen, were determined under various conditions. A volumetric process depending upon the use of potassium permanganate was employed for the estimation of the cuprous compound when it occurred together with the basic sulphate of copper and magnesium before mentioned.

The authors found in each case that they investigated that the sum of the magnesium equivalents of the cuprous oxide, copper, and hydrogen obtained agrees very closely with the amount of magnesium employed in the experiment. The magnesium is therefore proved to have displaced from the solution of cupric sulphate, substances which are chemically equivalent to it, though only a small and variable proportion of these substances consists of metallic copper. The authors have shown that the nature of the reaction is not influenced by the presence of slight impurities in the cupric sulphate employed by carrying out similar experiments with a specimen of the salt obtained by six successive re-crystallisations of a sample which was originally almost pure. Pickering's observation of the formation of a basic sulphate of copper by the decomposition of a solution of cupric sulphate by boiling, has been incidentally confirmed, but the formula which the authors attribute to this compound is $4\text{CuSO}_4 \cdot 7\text{Cu}(\text{OH})_2 \cdot \text{H}_2\text{O}$.

NOTE—24th November.—The authors have now referred to the paper by Commaille (*C. R.*, 1866, lxiii., 556), and they find that an equation is given which is supposed to represent the reaction between magnesium and cupric sulphate solution. This equation, when it is put into modern form and corrected for an obvious error, reads as follows:—



No details are given as to the temperature or the strength of the cupric solution which acted on the magnesium, nor are the analytical data stated upon which the above equation is founded. It will be noticed that the formation of basic magnesium compounds is not accounted for in the above equation. The authors consider that the results of their own analyses and examination prove that the composition of the basic sulphate of copper is variable, but that it does not correspond to the above formula, and certainly occurs in the condition of hydroxysulphate. They have never found that the proportion between the quantities of cuprous oxide and metallic copper is such as can be definitely represented by means of an equation, but rather that it varies within wide limits under different experimental conditions.

It will therefore appear that Commaille's imperfect study and statement of the reaction may well be supplemented by a more complete investigation.

The authors desire to place on record the fact that, when zinc acts upon a cold solution of copper sulphate, small bubbles of gas are evolved; and that when a hot solution of cupric salt is employed, an appreciable quantity of hydrogen can be collected, and cuprous oxide is found in the residue. They intend to study this reaction in detail.

DISCUSSION.

Professor TILDEN remarked that the work of the authors had been anticipated by the experiments of Commaille, who had published thirty years ago the results of an enquiry into the action of magnesium on neutral metallic salts, including copper sulphate.

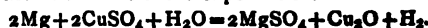
When magnesium is immersed in an aqueous solution of copper sulphate free from acid, the action seemed to take the following course:—First, there was a precipitation of spongy metallic copper, which, in contact with the magnesium, gave a couple capable of decomposing water at common temperatures. Hydrogen was then evolved, and a crust of magnesia formed on the surface of the metal. The copper salt was locally reduced by the hydrogen to the cuprous state, and this, in the presence of the magnesium oxide, led to the precipitation of cuprous oxide and of a basic cupric salt. The green precipitate formed therefore consists of magnesia and basic cupric salt, the proportion varying according to the temperature and strength of the solution. This hypothesis is borne out completely by the results both by Commaille and the authors of the paper, and serves to account for the apparent inactivity of the magnesium which is mechanically protected by the crust which forms upon its surface. The speaker had tested the suggestion that the metal was rendered inactive by a film of hydrogen and had come to the conclusion that this was not the case.

Professor CLOWES said that the origin of the investigation dated back some two years, the irregular action of magnesium on the cupric solution having been noticed in the course of experiments made in connection with a laboratory curriculum which he was then drafting for elementary students. It was anticipated that the reaction of magnesium on cupric sulphate might serve to establish the relative chemical values of copper and magnesium, but this was found to be impracticable owing to the evolution of a large amount of hydrogen. If such irregular actions occurred, instead of the simple replacement of one metal by another in solutions of its salts, it would seem that this method of determining chemical equivalents was not of general application.

Mr. CAVEN, in reply, said he regretted that he had overlooked the paper by Commaille on this subject to which Dr. Tilden referred.

It seems improbable that cuprous salt is formed by the reducing action of nascent hydrogen upon the cupric salt in the solution. This would lead to the formation of free sulphuric acid, which would decompose the cuprous oxide formed at the commencement of the reaction into cupric sulphate and metallic copper. If nascent hydrogen reduces the cupric salt at all, it can only produce metallic copper. Sulphuric acid would be liberated in this case also, though not in immediate contact with cuprous oxide. It may be that the process of solution of the magnesium hydroxide and basic copper salt which occurs at the close of the reaction in concentrated solutions, depends upon the action of the free acid thus produced.

The authors suggest that the magnesium reduces the cupric solution directly, producing cuprous oxide and hydrogen in the following manner, the proportion of the hydrogen evolved in the gaseous state being dependent upon the conditions of the experiment:—



Some such reaction as this, proceeding simultaneously with the action of the couple, may account for the immediate formation of cuprous oxide, and the vigorous evolution of hydrogen at the moment of immersion of the magnesium. The action of the magnesium-copper couple on the water is not in itself sufficient to account for the very large quantity of hydrogen which is obtained, or for the rapidity with which it is produced, since, when the couple acts upon pure water, the hydrogen is evolved only very slowly. This view of the origin of the cuprous oxide is borne out by the fact that it occurs, together with metallic copper, quite from the commencement of the reaction, and that the formation of hydrogen appears to begin directly the magnesium is plunged into the solution. This would not be the case if the formation of a metallic couple were necessary for the evolution of the gas.

There is no cessation in the evolution of gas during the course of the reaction under any circumstances. The

length of time necessary for the completion of the reaction in dilute solutions is undoubtedly due to the mechanical protection of the magnesium by the deposit, as Professor Tilden suggested.

*122. "Properties and Relationships of Dihydroxy-tartaric Acid." By HENRY J. HORSTMAN FENTON, M.A. Bearing in mind the highly interesting constitution of hydroxytartaric acid, and the close relationship which has been shown to exist between this acid and dihydroxy-maleic acid, a further investigation of its properties appeared to be desirable, especially as the free acid appears to have been scarcely studied.

It is now found that the free acid may very easily be prepared in a pure state by oxidation of dihydroxymaleic acid in presence of water. The yield is over 70 per cent of that demanded by theory. The dry acid shows no tendency to lose water at 90°. In aqueous solution, the acid decomposes when heated into tartronic acid and carbon dioxide. This reaction affords a very convenient method for the preparation of pure tartronic acid, the yield being 97 per cent of that theoretically obtainable. On titration with alkalis at 0° dihydroxytartaric acid behaves normally as a dibasic acid. But the results obtained at the ordinary temperature with caustic alkalis, using phenolphthalein as indicator, are considerably higher, being intermediate between those required for a di- and a tri-basic acid. These high results are shown to be due to the partial decomposition of the salts formed into tartrates and carbon dioxide.

By careful treatment with certain reducing agents dihydroxytartaric acid may be reduced to dihydroxymaleic acid or its isomeride. Zinc, in calculated quantity, and dilute acid reduces it to the β-form. Hydrogen bromide reduces it to dihydroxymaleic acid (α-form) with liberation of free bromine.

The reaction $C_4H_4O_6 + 2H_2O + Br_2 = C_4H_6O_8 + 2HBr$ is in fact a reversible one, the final distribution depending upon the masses of the reacting substances, and perhaps somewhat on the temperature.

*123. "The Molecular Association of Liquids and its Influence on the Osmotic Pressure." By HOLLAND CROMPTON.

In a paper which I recently had the honour of bringing before this Society (*Trans.*, 1897, lxxi., 925), I contended that the molecular association of liquids exercises an influence on their osmotic pressure, and endeavoured to show how this influence may be taken into account. My attention has been drawn to the fact that Planck, more especially, has long since proved that association could have no effect on the osmotic pressure of liquids (see the discussion between Planck and Wiedemann, *Zeitsch. Physik. Chem.*, 1888, ii., pp. 241, 343). This being the case, I wish to offer the following brief criticism of Planck's results.

In his "Vorlesungen über Thermodynamik" (Leipzig, 1897), Planck deduces, on p. 235, the following formula for the osmotic pressure of a dilute solution:—

$$P = \frac{R\theta}{n_0 m_0 v} (n_1 + n_2 + n_3 + \dots).$$

Here R is the constant of the gas equation, θ the absolute temperature, $n_1 + n_2 + n_3 + \dots = n$ the number of dissolved molecules, n_0 the number of molecules of the solvent, m_0 its molecular weight, and v its specific volume (volume of unit weight). Since $n_0 m_0 v = V$ is practically the whole volume of the solution $P = R\theta n/V$. This is, of course, van't Hoff's equation for the osmotic pressure, and it is argued that the product $n_0 m_0 v$ being simply the total volume of the solution, the pressure is independent of any change produced by association in m_0 . In other words, as long as V is constant P will remain constant, no matter what the value of m_0 .

The nature and derivation of this relationship will perhaps be best understood if we suppose that we have two gases, and that n molecules of the first are dissolved in,

or mixed with, n_0 molecules of the second. The partial pressure of the first gas in the mixture is $n/(n_0 + n)$ of the total pressure, or if n is small in comparison with n_0 , we may say n/n_0 of the pressure of the second gas. The pressure of this latter is RT/V_0 , and, in order that R may have a fixed value, let V_0 be the molecular volume $m_0 v_0$ of the gas, where m_0 is its molecular weight, and v_0 its specific volume. The partial pressure of the dissolved gas then becomes $p = RTn/n_0 m_0 v_0$, or, since $n_0 m_0 v_0 = V_0$, whole volume of the solvent gas, $p = RTn/V_0$.

These equations for the partial pressure of a gas present in a dilute state in any gaseous mixture are identical in form with those of Planck for the osmotic pressure of dilute solutions. It follows, then, that we have apparently only to liquefy the solvent gas, to substitute the molecular volume of the resulting liquid solvent for that of the gaseous solvent, and the partial pressure of the dissolved gas then becomes its osmotic pressure in the solution.

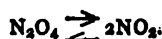
The result is not surprising, for it is obtained by a practical reversal of Planck's line of argument. It will be found (*loc. cit.*, p. 215) that, in his treatment of the thermodynamics of dilute solutions, he adopts the view that any such solution could be completely gasified without any change in the values of n and n_0 , and, of course, the osmotic pressure of the dissolved compound in the solution would then become the partial pressure of the dissolved gas in the gaseous mixture. But the view that a solution could be completely gasified without any change of a permanent character in the values of n and n_0 cannot be regarded as justifiable in the light of all the recent work on the molecular condition of liquids. And although it is not surprising to find that, starting with this view, Planck subsequently comes to the conclusion that association of the solvent does not influence the osmotic pressure of a solution, the reasoning evidently moves too completely in a circle to carry much conviction with it.

For, in order to ascertain what effect a change due to molecular association in the solvent would have on our formulae, let us first take the case of the mixed gases, and suppose the gas we term the solvent to be one, say, like nitrogen dioxide, NO_2 , which undergoes association as the temperature falls. Such association will bring about a change of m_0 to xm_0 , of n_0 to n_0/x , and of v_0 to v_0/x , if x represents the degree of association (factor of association) for any temperature T. The partial pressure of the dissolved gas now becomes $p = xRTn/n_0 m_0 v_0$, or since $n_0 m_0 v_0 = V_0$, $p = xRTn/V_0$. Hence, although when a gas is dissolved in any other gas, and is present only in a dilute condition, its partial pressure will, under normal conditions, follow the law that $pV_0 = nRT$ if association of the solvent sets in, the law for perfect gases no longer holds, but the relationship becomes $pV_0 = xnRT$ where x represents the factor of association of the solvent for the temperature T.

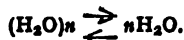
In fact, the partial pressure of the dissolved gas is, under the above conditions, dependent on the volume occupied by the solvent. Under normal conditions the solvent obeys the usual gas equation, and the partial pressure therefore alters in accordance with this relationship. But if association or dissociation of the solvent sets in, the gas equation will not immediately apply, but will require a suitable modification, and this modification will hold also for the partial pressure formula.

Now the osmotic pressure of a dilute solution presents an analogous case. As long as the solvent is a monomolecular one we may accept Planck's reasoning, and hold that the osmotic pressure follows the law $P = nR\theta/n_0 m_0 v$. But suppose that the solvent begins to undergo association, so that for any given temperature θ a change of m_0 to xm_0 has taken place. At the same n_0 changes to n_0/x , and a simultaneous change will be found to have taken place in v . This last change has, it seems to me, been entirely overlooked up to the present, although any one who gives the matter a moment's consideration cannot fail to admit that an associated liquid would not have the density of the same compound in the

monomolecular condition. All the evidence at present points to the conclusion that association increases the density of a liquid, and that consequently association would alter the value of v to v/a . The precise magnitude of a cannot at present be ascertained (unless perhaps with the aid of Traube's investigation of the molecular co-volume), but its value is probably not far removed from that of x . The osmotic pressure, then, of any dilute solution, the solvent of which is undergoing association, would be given by $P = \pi n R \theta / n_0 m_0 v$, or, since $n_0 m_0 v = V$, by $P = \pi n R \theta / V$, an expression that is similar to the partial pressure formula. While, then, the osmotic pressure of any substance in solution in a monomolecular solvent follows the gas equation $PV = R\theta$, if the solvent is one which is undergoing association or dissociation, the equation for the osmotic pressure becomes $PV = aR\theta$. And here another point which has apparently always been overlooked by those who contend that association of the solvent does not influence the osmotic pressure may be alluded to. It has been usual, hitherto, to compare with one another two stable systems, one of which is assumed to contain a monomolecular solvent, and the other the same solvent in the associated condition, and to show that the gas equation $PV = R\theta$ applies to each. The comparison, however, should not be between a monomolecular and an associated solvent, but between a monomolecular and an associating solvent, *i. e.*, one in which the association is continually altering with the temperature. The gas equation would hold for the gas NO_2 , and it would hold equally for the gas N_2O_4 , but it will not apply throughout the range of temperature in which—



The gas equation might in like manner hold for the osmotic pressure of a substance in solution in water of the molecular weight 18, and it might also hold for water of the molecular weight 3×18 , but water, as we know it, is neither of these compounds. It is a substance the association of which is continually changing with the temperature, and the attempt is therefore being made to apply the gas equation in the range of temperature throughout which—



Van Laar has, like Planck, treated the osmotic pressure of dilute solutions from the thermodynamical standpoint, and confirmed the latter's conclusion that association of the solvent does not influence the osmotic pressure. I quote here literally what Van Laar says on this matter (*Zeit. Physik. Chem.*, 1895, xviii., 274):—

"We found that the osmotic pressure $\pi = R\sigma(x+a)/v_a'$, where $\sigma(x+a)$ has the value Σc_i ,* and v_a' , the volume of 1 grm. molecule of the pure solvent. For water v_a' was therefore given the value 18. If, however, association occurs, the weight of the grm. molecule would be $18a$, so that for v_a' the volume of $18a$ grms. of water must be substituted. Calling this last v_a' , then $V_a' = av_a'$. But also σ , the concentration of the dissolved substance, becomes a times greater. For we have now no longer one molecule of the dissolved substance to n molecules of water (taking the mol. wt. of water = 18), but one molecule to n_1/a molecules of water, so that the concentration must be expressed by an a times greater number than before. Calling this C , then as $C = a\sigma$, the expression—

$$\pi = RxC(x+a)/V_a'$$

does not differ from $\pi = R\sigma(x+a)/v_a'$, where the quantities v_a' and σ are independent of the association."

The italics in the above are mine, for it is to this portion of the paragraph that I wish to direct attention more especially. The molecular volume of any compound is given by M/d , where M is the molecular weight of the compound and d its density. Now Van Laar supposes

that if liquid water had the molecular weight 18, its molecular volume would be $18/x$; or if it had the molecular weight $18a$, its molecular volume would be $18a/x$. In other words, the same density is to be assigned indiscriminately either to the monomolecular or to the associated compound. But can any one doubt if liquid water were obtainable having the molecular weight 18, that it would most certainly not have the density x at ordinary temperature? Take water merely as the first term of the $\text{C}_n\text{H}_{2n+1}\text{OH}$ series of alcohols, a series the members of which are themselves associated, but not to the same extent as water, and analogy at once points to a much lower density than x for monomolecular liquid water, a density that is in fact probably not far removed from $1/a$. In fact there is absolutely no evidence that the molecular volume of an associated liquid does differ greatly from that of the same liquid compound in the monomolecular state. Van Laar's argument *against* is converted therefore into an argument *for* the effect of association on the osmotic pressure.

PHYSICAL SOCIETY.

Ordinary Meeting, December 10th, 1897.

Mr. SHELFORD BIDWELL, President, in the Chair.

Mr. ALBERT CAMPBELL exhibited (1) An experiment to illustrate alternate exchange of Kinetic Energy. Two brass spheres, each about 1 inch diameter, are suspended from the same point by equal wires. One of them is then thrown so as to describe a circular orbit. The second sphere, starting from rest, gradually takes up motion from the first sphere, and in turn describes a circular orbit. The first now comes to rest, and the reverse process takes place. This alternating action repeats itself until all the energy is lost in the wires. (2) An experiment to illustrate the Low Heat-conductivity of Glass and the Expansion of Glass by Heat. A long tube is clamped at the lower end, in a vertical position. One side of it is then heated with the flame of a Bunsen burner, and the glass is observed to bend, moving over a fixed mark near the top of the tube. When the flame is withdrawn, the first position is quickly regained.

Mr. CAMPBELL then read a paper on "Temperature Compensators for Standard Cells."

Some account of the methods adopted by the author has already been published; he now describes the apparatus. The first compensating arrangement (3) can be used for keeping the potential-difference between two points of a conducting system constant at all room-temperatures. Or it can be adapted to modify the voltage of a standard cell to some convenient whole number. This arrangement (3) resembles a Wheatstone's bridge with the galvanometer-branch removed. One pair of opposite arms is of copper, the other pair is of manganin. The bridge-battery is a Leclanché cell: this supplies the auxiliary voltage, which is utilised at the two galvanometer-points of the bridge, and is there applied in series with the standard cell. In an alternative method, suggested by Mr. C. Crawley, only one of the four arms is made of copper. The second compensating arrangement (4) is intended to maintain constant potential between two points, at all room-temperatures. For this purpose two wires, a and b , are connected in parallel. One of them, a , is all of manganin; the other, b , is partly copper and partly manganin. Constant current is applied at the ends of a and b . The various resistances are chosen so as to give constant difference of potential between the ends of the manganin portion of b . By this method the potential difference can be maintained to within 1 in 2000.

Mr. SWINBURNE said that twelve or thirteen years ago he had given a good deal of thought to compensation by wires of different temperature-coefficients. The first thing he tried was a Wheatstone's bridge. This was

* The v here is the v of the Planck formula.

compensated by making the bridge-arms of wires whose temperature-coefficients differed—as, for instance, platinoid and copper. He then applied the same principle to the compensation of standard cells, using a potentiometer method that gave direct readings, and to the compensation of voltmeters and Watt-meters. These results were published between 1885 and 1890, in the electrical journals. He believed that Mr. Evershed had also developed this idea, by putting “back” turns on voltmeters, and by other differential devices. The details of Mr. Campbell's apparatus had a few points of special interest. The way in which he connected up the bridge (3) seemed particularly worthy of notice.

Prof. AYRTON asked whether thermo-electric effects produced difficulty in the compounded arrangement.

Mr. CAMPBELL said the system was symmetrical, and the thermal currents were consequently neutralised.

Mr. APPELBY, referring to experiment (2), said it was identical with one that had been shown for the past eight years at lectures at Cooper's Hill College. It was specially interesting as illustrating the deflection that occurs with girders and bridges when exposed on one side to sunshine.

Mr. J. ROSE-INNES read a mathematical paper on “Lord Kelvin's Absolute Method of Graduating a Thermometer.”

Lord Kelvin has investigated the cooling effects exhibited by various gases in passing through a porous plug. He found that for any gas, kept at the same initial temperature, the cooling effects were proportional to the difference of pressure on the two sides of the plug. He also found that, for any one gas, the cooling effect per unit difference of pressure varies approximately as the inverse square of the absolute temperature. This rule holds very well in the case of air; it is not so satisfactory for carbonic acid; it fails for hydrogen. With hydrogen there is a heating effect that increases, if anything, when the temperature rises. Mr. Rose-Innes proposes an empirical formula, containing two disposable constants, α and β , characteristic of the gas in question. Denoting by T the absolute temperature, he finds that, very approximately, the cooling effect is given by the expression—

$$\left(\frac{\alpha}{T} - \beta\right).$$

This relation includes the three cases—air, hydrogen, carbonic acid—under one form, and thus enables them to be treated in one common investigation. Moreover, the differential equation concerned in the thermo-dynamic scale is thereby rendered more manageable,—it leads to simple algebraic results after integration. The paper discusses the thermo-dynamic correction for a constant-pressure gas-thermometer, and the correction for a constant-volume gas-thermometer; also an estimate of the absolute value of the freezing-point of water: the results obtained take, for the most part, a very simple shape, using the above expression for the cooling.

Dr. S. P. THOMPSON said the empirical expression,—

$$\left(\frac{\alpha}{T} - \beta\right),$$

indicated that at some particular temperature the cooling effect vanished; that was a point suggestive of useful results if investigated by experiment.

Mr. J. WALKER read a communication from Mr. BAYNES on the paper, and remarked on the desirability of adopting two constants. He thought that further experiments should be made to discover how specific heat at constant temperature depends on temperature. The calculated values for hydrogen were too few to be taken as evidence of the validity of the rule.

Mr. ROSE-INNES, in reply, said that from what was known of hydrogen, it might be expected to behave at ordinary temperatures as air behaves at higher temperatures. His object was, if possible, to include in one

formula the case of the three investigated gases. This was much better than having a separate formula for each gas. Whether or not hydrogen was conformatory with air and carbonic acid might be considered as *sub judice*; it required further experimental data to test the formula in that case.

The PRESIDENT proposed a vote of thanks to the authors, and the meeting was adjourned until January 21st, 1898.

SOCIÉTÉ D'ENCOURAGEMENT POUR L'INDUSTRIE NATIONALE.

November 26, 1897.

M. MASCART, President, in the chair.

The SECRETARIES read several letters received, after which M. GROIGNARD exhibited an automatic valve for the arrest of noxious fumes, and an automatic water level control-valve of similar construction.

M. A. POUCHET exhibited an apparatus for aerial navigation.

Several gentlemen were proposed as candidates for the position of member of the committee on Mechanics, and two candidates were announced as having been elected members of the Society.

An interesting paper was then read by M. BECHMANN on the “Purification of the Seine,” for which he received the thanks of the Society. This paper will be printed in the *Bulletin*.

The next meeting for the election of officers for 1898 was announced to take place on December 10, 1897.

NOTICES OF BOOKS.

A Complete Catalogue of Practical Physical Apparatus Manufactured by W. J. GEORGE, late BECKER & Co.

THIS catalogue contains a complete list of the Physical apparatus recommended by Professor Schuster and Dr. Lees in their text-book on “Practical Physics,” and will doubtless be found to be of great assistance to both teachers and students not already provided with the necessary apparatus for their studies. It is a great advantage and saving of time to be able to order any particular piece of apparatus which may be required, with a knowledge that it is kept in stock and can be delivered at short notice, thus avoiding those tiresome delays to which we are too much accustomed. The list appears to be comprehensive, well illustrated, and conveniently arranged.

OBITUARY.

DR. CAMPBELL MORFIT.

By the death of Dr. Campbell Morfit, M.D., formerly Fellow of the Chemical Society and of the Institute of Chemistry, which took place on the 8th inst. at South Hampstead, the science of applied chemistry has suffered a severe loss. An American by birth, he had for many years past been a London resident. In 1854 he was appointed Professor of Applied Chemistry in the University of Maryland, and was one of the scientific advisers of the United States Government previous to the Civil War.

He was the author of several standard works on industrial chemistry, including “Chemical and Pharmaceutical

Manipulation," "Arts of Tanning and Currying," "Oleic Soaps," and with Dr. James C. Booth was joint editor of the "American Encyclopedia of Chemistry."

His work included researches on the guanols, salts, sugars, analyses of coals, gum mesquite, and glycerin. During the latter period of his life his attention was devoted to the improvement of technical processes, the preparation of condensed food rations, the manufacture of paper, and in especial to the questions of the refining of cotton and linseed oils and the utilisation of cotton-seed oil as a food substance.

His loss will be deeply felt by a wide circle of scientific and literary friends.

CORRESPONDENCE.

SEPARATION OF NICKEL AND COBALT FROM IRON.

To the Editor of the Chemical News.

SIR,—May I take exception to the opening statements of the article on the separation of Ni and Co from Fe, &c., which you reprint in the CHEMICAL NEWS (vol lxxvi., p. 279).

To say that there is no satisfactory method known for effecting the separation of Ni and Co from large quantities of Fe is surely untrue.

The following, if test analyses are to be credited, are a few references to satisfactory methods already described in your journal:—

1. Precipitation of the iron with lead oxide (Field, CHEM. NEWS, i., 4).
- 2 and 3. Precipitation as ferric phosphate in acetic solutions (Moore, CHEM. NEWS, liv., 300; Campbell, lxi., 139).
4. Treatment with excess of soda pyrophosphate; whereby, even in presence of much iron, a clear colourless solution is obtained, which is subsequently titrated with cyanide of potassium (Moore, CHEM. NEWS, lix., 292).
5. Conversion of the iron into ferricyanide, Ni into nickelocyanide, and subsequent precipitation of Ni by a solution of bromine in potash (Moore, CHEM. NEWS, lvi., 3).

This is not a complete list of the processes to be found in the CHEMICAL NEWS. Other well-known and largely used processes are to be found in text-books specially devoted to steel analysis. Two other processes which have been well tested and find great favour are Campbell and Andrew's xanthate process (*Journ. Amer. Chem. Soc.*, Feb., 1895), and Herschell's hydrate process ("Fresenius's Quantitative Analysis," p. 437).

The second paragraph specifies hydrate and basic acetate precipitates as "always containing a considerable proportion of other metals (Ni, Co, Mn, Cu, &c.)."

The case for acetate precipitations has already been presented at sufficient length. It is necessary only to refer to the separate papers—Nickel, p. 49; Cobalt and Manganese, p. 165; Copper, p. 210—to see that respecting acetate the charge is unjustly made.

It is not claimed that any of the above processes are more accurate than the electrolytic process, although some of them, with their accompanying estimations, are equally so. But when the employment of the process in everyday practice comes to be considered, their superiority in point of quickness and general convenience is most marked.—I am, &c.,

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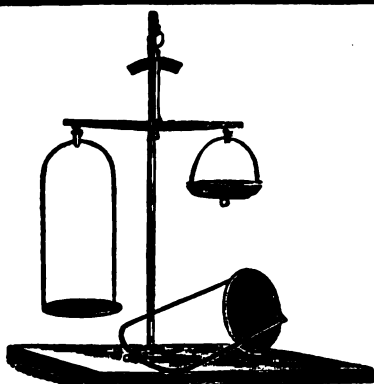
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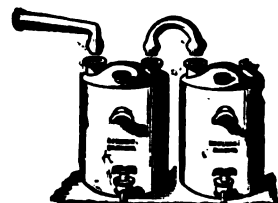
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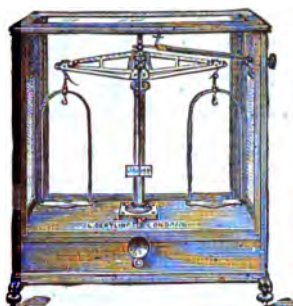
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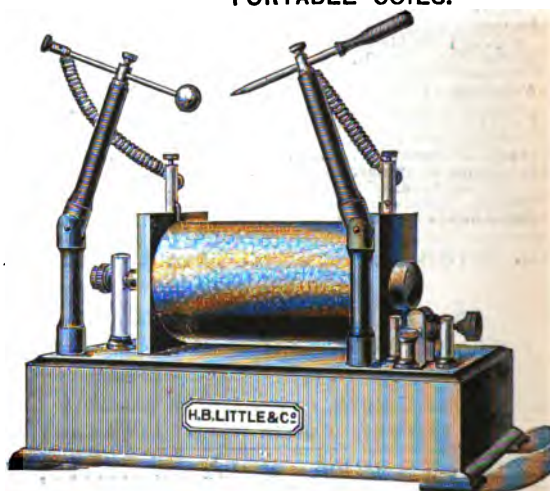
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THE ESTIMATION OF COPPER IN PRESENCE OF OTHER ELEMENTS.

By HARRY BREARLEY.

(Concluded from p. 293).

Iron.—Precipitated with either alkali, the chief objection hitherto has been the colour of its precipitated hydrate. With soda the colour is less objectionable. As the cyanide is added the precipitate becomes redder, and something may be deduced from watching the changing colour contrasts as the cyanide falls into the solution. There has been some question as to how this element should be treated. Some are all for filtering it off, while others object that the ammoniacal copper solution and ferric hydrate cannot be completely separated even by re-precipitation or washing. Indeed it is claimed that its presence "is rather an advantage, as it acts as an indicator to the end of the process" (Sutton, "Volumetric Analysis," p. 146). It must certainly be filtered off before the AgI end reaction can be applied. It should be pointed out, however, that then the above objections apply to the titrated solution no longer, because it already contains, if need be, more than enough cyanide to decolourise the copper solution, and is thus more likely to avoid any error due to the presence of the precipitate than, when the colour only is relied on, stopping the titration when the liquid only is barely decolourised.

It will be noticed that the ammonia series gives better results than the soda series. The following suggestion covers only a small error, but as it applies to many elements it is placed against this noted difference.

It would need some ammonia to precipitate the iron in those solutions containing it. This would decrease a given excess of ammonia, and increase the amount of ammonium chloride. Decreased ammonia means increased cyanide, and increased ammonium chloride acts in the same direction, and therefore any imperfect recovery is partially counterbalanced. This is not given as a complete explanation of the difference between the values of the ammonia and soda series, but only as a circumstance which favours increased accuracy in the one and is indifferent to the other.

Citric or tartaric acid are less applicable here than before, on account of the coloured ferric solution. Pyrophosphate is said to give, "even in the presence of much iron, a perfectly clear, colourless solution" (Moore).

Manganese.—There is finally a precipitate in each case. With ammonia it formed only while the cyanide was being added. After completing the cyanide reaction and filtering off, a slow precipitate of manganese may occur and confuse the end reaction. With soda the white precipitate changes immediately on adding cyanide. No further precipitation was noticed after filtering.

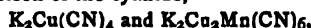
Has the cause of these low results ever been explained? Field suggested that a colourless compound of the two metals existed, and tried, but vainly, to prepare an ammoniacal cyanide of manganese and copper. There has been no new attempt made during this investigation to separate such a double cyanide from the titrated liquid, but it is noticeable that such a compound as Field sought to prepare has since been shown to exist, and may account for the low results.

The compound in question is potassium cupro-manganocyanide, $K_2Cu_2Mn(CN)_6$, which may be prepared, according to Straus (*Journ. Chem. Soc. Abs.*, 1895, i., 485), by adding manganese acetate to a solution of potassium copper

cyanide $(KCN)_6(CuCN)_2$; it crystallises in cubes, and is stable only when damp.

On adding ammonia to a solution of a manganese salt we have a hydrate precipitated which is eager to oxidise itself, as is seen by the changing colour. In our copper solution such a precipitate may be considered as a reducing agent with power to prepare a cupro-cyanide for the already unoxidised manganous salt, and so to form the cupro-manganocyanide.

A comparison of the cyanide,—



shows that the latter requires less CN per atom than the former, and therefore may account for the low result.

To turn to the more congenial task of describing a means of avoiding the interference. In attempting, a year ago, to titrate the copper separated from a mixed solution of copper and Swedish bar-iron, it was found that very small quantities of something formed a cloud black enough to hide the fading copper colour. Without suspecting that what seemed so insignificant an amount of manganese (0.1 per cent) was the cause of the trouble, it was found that if the solution was neutralised, made faintly acid, and the KCN added, there was no precipitate whatever formed. That was with ammoniacal solutions.

A similar procedure may be applied to the soda titration as follows:—To the acid solution of copper and manganese, add soda carbonate until a small precipitate forms, dissolve in a slight excess of acid, add cyanide, and then the usual excess of soda carbonate. There are no distinctive colour changes, and therefore it is necessary to know beforehand the approximate amount of copper present. On adding the carbonate, the manganese falls as a white and somewhat crystalline precipitate of considerable bulk, but on standing it becomes brownish, granular, or powdery, and more compact.

The following results were obtained in this way:—

0.0	0.01	0.05	0.10 grm. Mn present.
0.1000	0.1000	0.1004	0.1010 Cu found.

The precipitate should not be filtered off until the above physical changes have taken place, otherwise a precipitate may form in the filtered solution and confuse the iodide turbidity. The chemistry of this changed physical state has not been studied. When a bath colour has been reached, there is no further darkening on prolonged standing. The filtration is very easy.

Chromium: Soda.—A precipitate formed which is somewhat soluble in the excess of soda carbonate, and hence the filtered solution is green. This colour led Thomson to conclude that it was impossible to perform the titration in presence of chromic salts; but it is only one of a number of cases where the presence of large amounts of carbonate in his cyanide introduced interfering circumstances.

With the AgI indicator the titration is not satisfactory, because in the first place the colour reaction cannot be utilised, and, further, it is believed that the chromium, like the zinc precipitate, but in a less degree, can retain some of the copper in presence of an excess of cyanide.

Ammonia.—A precipitate which is somewhat soluble in the alkali, but with the excess used (10 c.c. 2 normal) the filtrate is quite colourless.

The tabulated results were altogether unexpected. It will be noticed that Field registers "no interference," but by what procedure he arrived at so variant a result it is impossible even to conjecture. The ammonia titration has been repeated again and again, and always with similar results.

Such a wide variation could not be caused by dissimilar indicators; besides, experiment shows that if the colour only be adhered to, the percentage error may be over 40 per cent.

The above results must be taken only as approximation, for this reason:—Each sample and the standard were treated with equal amounts of cyanide, and therefore

"0.05" contains a larger excess than the standard, and "0.10" a still larger. This would (if the influence of the excess of cyanide on the copper is the only consideration) make the results somewhat high; "0.10" more so than "0.05." A corrected result might even show the proportional action of increasing amounts of chromium which is suggested by the given figures. Elsewhere this source of error has been guarded against.

I am unable at present to propose a probable explanation of the unexpected results; but I am able to point out that the chromium existing as chromic acid exerts no material influence, and thence an obvious means of overcoming the difficulty.

The yellow colour of the chromate does not very greatly interfere with the colour observation. The change is from green to a pure yellow.

Cobalt, Nickel, Silver.—These were not done. They would interfere in a regular way, and if estimated by other means could be allowed for in titrating a mixture. Thomson found it impracticable to estimate copper in presence of large quantities of cobalt. With only colour changes to rely on, this is undoubtedly the case. Field states the interference to be 12 per cent. This only serves to confirm Thomson's statement, because the interference of cobalt is really as great as that of nickel. An explanation of how this 12 per cent was arrived at would be interesting. Thomson finds nickel to interfere 120.4 per cent; Field, 95 per cent. This difference, and some others, might be accounted for by supposing that the amount of alkali and alkali salts varied with each operator if it were not for the striking agreement in their values for silver—31.7 and 32.0.

Gold, platinum, and palladium are omitted on account of their cost. Thomson finds them to interfere 32.65, 19.25, and 62.45 per cent respectively. Such large interferences suggest the possibility of estimating them by some modification of the cyanide titration.

Tin.—Precipitate with either alkali; not easy to filter. The effect of stannous salts was not quantitatively measured; they cause low results.

Molybdenum (Soda Molybdate).—No precipitate in either case.

Arsenic (Soda Arsenate).—No precipitate in either case.

Antimony.—A precipitate with either alkali; easily filterable.

Bismuth (Nitrate).—White precipitate; moderately filterable.

Lead (Acetate).—Precipitate with either alkali; moderately filterable.

Mercury (ous).—Final precipitate grey; only slight in ammoniacal solution, more with soda; easily filtered.

Mercury (ic).—No precipitate in either case after titration. It is noticeable that the influence of mercury is fairly regular. Denigés, who has based so many delicate analytical methods on the $\text{AgI} + \text{KCN}$ reaction, has recently (*Journ. Chem. Soc., Abs.*, 1897, ii., 433) applied it to the estimation of mercury.

Uranium (Nitrate).—**Soda:** No precipitate. **Ammonia:** Yellow precipitate, which does not dissolve on adding the cyanide, unless the cyanide contains considerable carbonate; filters rather badly.

Separating and Estimating Copper.

Precipitation of copper as subsulphocyanide by sulphurous acid and a sulphocyanide is a well-known means of separating copper from other elements. Hugo Tamm (*CHEMICAL NEWS*, xxiv., 91; Crookes, "Select Methods," p. 264) uses a mixture of equal weights of sulphocyanide and soda or ammonia bisulphite, and claims by these means to separate copper from whatever substance may accompany it. As the above table does not include all substances whatever, and as no means are suggested for avoiding some of the interferences, I gladly draw attention to Tamm's reagent. So far as I have had occasion to use it, or rather its like (H_2SO_3 and KCNS), the separations have been very satisfactory. If the estimation of the

copper were as easy as its separation, all would be well, but at the very next operation it behaves badly by passing through the filter. The subsequent drying of the filter is lengthy, and thus an admirable separation is largely overlooked on account of the difficulties in making the estimation.

The following recommendations are made to meet this difficulty:—

Having precipitated the copper, decant the solution on to a small asbestos or paper pulp filter; if necessary, the filter may be washed with a dilute solution of the precipitating reagent and then replaced in the precipitating flask. Add a few c.c. of nitric acid, 20 c.c. 2N hydrochloric acid, and boil for a few minutes. The copper and any adhering sulphurous acid are oxidised, and the cyanogen compounds are decomposed. Cool, neutralise with soda carbonate, add the usual excess, and titrate as usual.

No test analyses are given. The analyses of nickel, copper, and zinc alloys, to be given in a subsequent paper, will serve this purpose equally well.

The Laboratory,
Norfolk Works, Sheffield.

THE EXACT WEIGHT OF OXYGEN, HYDROGEN, AND NITROGEN.

By the Author of "Reform of Chemical and Physical Calculations."

PROFESSOR ARMSTRONG said at the Chemical Society, March, 1896: "*A pure substance is, and must ever remain, an ideal conception; we should not speak of a substance as pure, when such a condition of matter is known to be unattainable.*" If this be true (and many eminent chemists endorse that sentence), how can we rely upon experiments which give, for instance, the weight of hydrogen with five decimals when we know that the hydrogen we can prepare is *not pure*? even filtered through a diaphragm of hot platinum, it shows a "compound" spectrum (*CHEMICAL NEWS*, vol. lxxvi., p. 170), and as hydrogen is the lightest substance known, every admixture must increase its weight.

The weight of oxygen can be, and no doubt is, determined more exact than that of any other gas; and if Lord Rayleigh's determination of the weight of 1 litre oxygen = 1.42952 grm., is taken as correct for the latitude of London, the experimental determination of hydrogen, 1 litre = 0.09001 grm., is more than 1/16 of the weight of oxygen, and so are the results found by all other investigators; 1.42952/16 is only 0.089345 grm. The difference between the experimental and the calculated value is 0.090010 - 0.089345 = 0.000665 grm. per litre H; and such a difference would be caused if 0.39 c.c. argon are mixed with 999.61 c.c. pure hydrogen, or 0.2 c.c. argon + 0.3 c.c. N mixed with 999.5 c.c. pure hydrogen.

If pure N has the relative weight 14, O being = 16, then a mixture of 999 c.c. N + 1 c.c. argon would be = 14.005,—the value found by M. Leduc's latest experiments. If chemists can *prove* that they are able to detect and remove these or other impurities, if they further can *prove* that their instruments and observations are absolutely correct, and that every experimenter gets the *same result*, then they may dispute the relative weights H=1, N=14, O=16.

C. J. T. HANSSEN.

Copenhagen, December 11, 1897.

The Astronomer-Royal of the Berlin Observatory, who has closely studied my "Reform," has authorised me to publish the following:—

(TRANSLATION).

Berlin, Sternwarte.

... My opinion is, that such chemical and physical investigations as are developed in your "Reform of Calculations," quite apart from the ordinary course of research and study, not only now are of high importance, but ultimately may lead to very great and eminently practical simplifications of scientific work and study, and therefore are worthy of public acknowledgment and promotion.

(Signed),

Professor WM. FORSTER, Dr. phil.

Note to Review, CHEMICAL NEWS, August 6th, 1897, page 70.—The "Carlsberg Foundation" is one of the principal institutions for the advancement of science in Copenhagen, and administrated by Professors of the University. My "Reform" is printed under the patronage and at the expense of that Institution, not by the Carlsberg Foundation, as stated in the CHEMICAL NEWS.

ERRATA.—CHEMICAL NEWS, November 26, 1897, p. 264, col. 1, instead of "0.42952" read "1.42952"; and instead of "56/10" read "56/80."

QUALITATIVE RESEARCH ON TRACES OF ALKALINE CARBONATES IN PRESENCE OF AN EXCESS OF BICARBONATES, OR OF BORAX.

By ALEX. LEYS.

FOR the purpose of differentiating between the bicarbonates and the alkaline carbonates we make use of a salt of magnesium, such as the sulphate, for example: the neutral carbonates give a white precipitate, while with bicarbonates the solution remains clear.

This reaction, which gives excellent results when applied to either one or the other of these salts, does not answer when they are mixed. Such is the case with certain products which are met with commercially, under the name of "milk preservers." In them we find mixtures of carbonate and bicarbonate of sodium, of carbonate of sodium and borax, or all three of these salts together.

From several analyses we have made we have found that with a mixture of crystallised bicarbonate and carbonate of sodium, in the proportion of 32 of the former to 68 of the latter, completely dissolved in water, no precipitate was formed with sulphate of magnesium, from which it might be concluded, relying only on this reaction, that the substance was formed of bicarbonate of sodium only. In the same manner a mixture of crystallised carbonate of sodium, with a sufficiently high percentage of borax, such as 40 of borax to 60 of the neutral carbonate, does not precipitate sulphate of magnesium, and leads one to believe in the same way, without further information, that the substance under examination is nothing but bicarbonate.

It is thus seen that a small quantity of bicarbonate or of borax may hide a considerable proportion of neutral carbonate. We have therefore sought for a more trustworthy and sensitive reaction. A saturated solution of sulphate of lime seems to fulfil our requirements.

Such a solution remains limpid during a certain time only when we add to it pure bicarbonate of soda; then we see gradually appear, throughout its mass, microscopic crystals which eventually make the liquid cloudy. With pure borax we also do not observe any cloudiness when the solutions are first mixed, and, further, the liquid does not become cloudy on standing; but as soon as the smallest quantity of neutral carbonate is present with one or the other salts in solution, the former, when added to a solution of sulphate of lime, immediately gives rise to a

heavy, opaque, white precipitate of carbonate of lime. This reaction is so sensitive that if we pour over a mass of commercial bicarbonate of soda, usually sold as pure, a quantity of water insufficient to dissolve it entirely, we immediately obtain with this first solution, when added to sulphate of lime, the white heavy precipitate characteristic of the neutral carbonate. If we throw away the first water, and add fresh, the second solution will often give the immediate precipitate, and it is only at the third addition of water that the presence of the neutral carbonate no longer shows itself, and that we usually get a solution of pure bicarbonate of soda.

It is easily seen from this, that if we use both sulphate of magnesium and sulphate of calcium, we cannot, when dealing with a mixture, be mistaken as to the nature of its constituents.

It will suffice, when the sulphate of magnesium does not give a precipitate, to try the sulphate of calcium reaction before coming to any conclusion. If, in the latter case, we do not obtain an immediate precipitate, then only can we be sure of the absence of the neutral carbonate.—*Journ. de Pharm. et de Chim.*, Series 6, vol. vi., No. 10.

ON THE SOLUBILITY OF AMMONIA IN WATER AT TEMPERATURES BELOW 0° C.

By J. W. MALLET.

ROSCOE and Dittmar's table* of the solubility of ammonia in water at different temperatures, under normal pressure, begins at 0° C., and there seems to be no recorded measurements of solubility at any lower temperatures. It is stated on the authority of Fourcroy and Vauquelin† that a "concentrated" aqueous solution of ammonia—the exact amount in solution not given; probably not determined—does not freeze till cooled to between -38° and -41°, that it then forms brilliant flexible needles, and that at -49° it solidifies to a grey gelatinous mass, almost destitute of odour.

It seemed interesting to try the effect of continuing to pass gaseous ammonia into an already strong aqueous solution at temperatures much below zero, and particularly to see whether any visible change of behaviour would mark the presence of enough ammonia to represent the hydroxide of ammonium, often assumed to exist in the ordinary solution, but without any surplus water. The proportion in question—one molecule of ammonia for one of water, might, from an inspection of Roscoe and Dittmar's table, be expected to occur at a temperature but little below zero. Taking advantage of a fall of finely pulverulent dry snow, followed by two or three days of cold weather, during which the atmospheric temperature was about -8° to -12° C., the following experiments were made last winter. In anticipation of such an opportunity, the apparatus had been made ready some time before.

A strong solution of ammonia in water was placed in a burette-like glass tube of the form shown in Fig. 1, of which the larger cylindrical part was about 220 m.m. long and 25 m.m. in internal diameter, the narrow tube below about 100 m.m. long and only about 1 m.m. in internal diameter, with a small glass stopcock about 30 m.m. above the orifice. The cylinder was graduated for about two-thirds of its length into cubic centimetres. By means of an india-rubber stopper, perforated to grasp tightly the narrow tube, and then cut in two in the line of its axis, this part of the burette was fixed in the neck of an inverted gas-jar, the body of which was about 180 m.m. high, with an internal diameter of about 120 m.m., so that the cylindrical body of the burette could be sur-

* *J. Chem. Soc.*, xii., 128—quoted in Roscoe and Schorlemmer's "Treatise on Chemistry," i., 385.

† Gmelin's "Handbook of Chemistry" (Cavendish Soc. transl.), ii., 425.

rounded by a freezing-mixture contained in the jar, without any leakage occurring at the neck, the narrow—almost capillary—tube and stop-cock being below and outside the jar. The open mouth of the burette above was loosely closed by a doubly-perforated stopper, through which passed a small tube for the introduction of ammonia gas, and an alcohol thermometer, each fitting so as to be easily slid up or down. The thermometer was carefully made by Mr. Henry J. Green, of Brooklyn, N.Y., contained pure (uncoloured) alcohol, was graduated on the glass stem, and had the (centigrade) scale verified by comparison with a (Fahrenheit) mercurial thermometer, itself tested by comparison with a gas thermometer, at the following points: $+32^{\circ}$, $+12^{\circ}$, -8° , and -28° F. The gaseous ammonia was obtained by simply heating in a flask the strong aqueous solution. There was, of course, no need for drying it, but it was cooled, and incidentally in large measure dried, by passing it first through an

graduation marks, from which a measured sample might then be taken off. A number of light glass flasks, each of about 300 c.c. capacity, furnished with stoppers, were in advance about three-fourths filled with water and severally weighed with accuracy on a good analytical balance. These flasks of water were brought down to 0° C. by immersion in snow, and—operating out of doors—the several samples of liquid ammonia were run into them from the burette, producing at once such large dilution that there was no further fear of loss of ammonia by standing over, with the stoppers inserted, until the flasks could be re-weighed—the gain of weight in each case representing the amount of liquid taken from the burette—and the amount of real ammonia determined by neutralisation with a standard solution of sulphuric acid. The first sample was taken at -40° C., and then, by simply removing the freezing-mixture of snow and hydrochloric acid from the jar, the temperature of the liquid in



FIG. 1.

empty flask surrounded by snow, and then through a spiral coil of glass tubing immersed in a bath of snow and common salt. Thence by an india-rubber connecting tube it passed into the already strong solution in water contained in the burette. This was surrounded in the jar by a freezing-mixture of snow and commercial hydrochloric acid, both materials having been separately cooled down in advance by snow and salt. In consequence of the evolution of heat as more ammonia was absorbed by the water in the burette, it became necessary to allow ample time for cooling down again, and to renew the freezing-mixture in the jar, drawing off the old mixture by means of a specially arranged and rapidly acting syphon. In order to get the lowest temperature reached (between -45° and -46° C.) it was necessary to doubly cool the last portions of snow and hydrochloric acid, first by snow and salt, and then by separate exposure to some of the already cooled snow and hydrochloric acid mixture. The passage of the gas into the solution was continued until it went through very freely unabsorbed, the liquid in the burette continually increased in volume, so that, when samples were drawn off by the stopcock, enough was first run off to effectually cool the projecting part of the narrow tube and the stop-cock itself, and to bring the level of the remaining liquid down to one of the

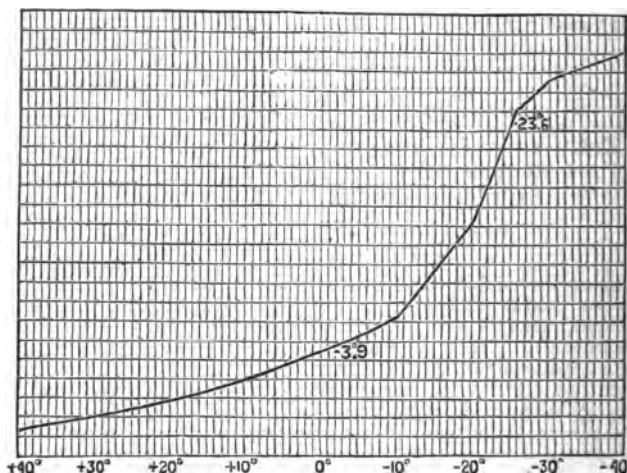


FIG. 2.

the burette was allowed to slowly rise, and as the thermometer marked different desired points it was raised for a moment out of the liquid, another sample drawn off into the water in one of the flasks beneath, and the thermometer lowered again. One or two fairly good measurements of the volumes of the samples were made, but, owing to the necessity for quick work, and the difficulty of seeing satisfactorily through the dimmed surface of the glass, most of these measurements were little to be relied on.

The following results were obtained, so expressed as to form a continuation of Roscoe and Dittmar's table. The atmospheric pressure at the time was 743–744.5 mm., corrected for temperature of the mercurial column.

At -10° C. 1 grm. of water dissolved 1.115 grms. of ammonia.

At -20° C. 1 grm. of water dissolved 1.768 grms. of ammonia.

At -30° C. 1 grm. of water dissolved 2.781 grms. of ammonia.

At -40° C. 1 grm. of water dissolved 2.946 grms. of ammonia.

The taking up of enough ammonia to form ammonium hydroxide was not attended with any apparent change of behaviour, and continuous liquefaction went on smoothly

to the lowest temperature reached, with no separation of any solid product. After the freezing-mixture had been withdrawn the temperature rose at first very gradually in consequence of rapid surface evaporation of ammonia. At -25°C . (barometer at 743.4 m.m., corr.), steady but not tumultuous ebullition took place and continued for several minutes. A sample taken at this point had the composition: 1 grm. of water and 2.554 grms. of ammonia.

By interpolation from Regnault's results, the tension of the vapour from liquefied ammonia at -25.6°C . would be about 1167 m.m.

A sample was also taken at -3.9° , which proved to consist of 1 grm. of water and 0.947 grm. of ammonia—this last corresponding almost exactly with the calculated proportion for ammonium hydroxide, and agreeing also quite closely with an extension of Roscoe and Dittmar's table to the temperature named, allowance being made for the pressure being a little below normal.

These results are graphically plotted in fig. 2, which includes also the determinations of Roscoe and Dittmar for $+40^{\circ}$ to 0° . It will be seen that the curve changes rapidly a little below the temperature corresponding to the formation of $(\text{NH}_4)\text{OH}$, and the amount of ammonia absorbed is much greater than would be called for by an extension of Roscoe and Dittmar's numbers.

As regards the reversal of curvature before -30° is reached, some allowance must doubtless be made for loss of ammonia by the samples drawn at the lowest temperatures in dropping through the air into the ice-water in the flasks in spite of the precautions taken to minimise loss from this source. And, in view of the fact that liquefied ammonia boils at -33.7° under a pressure of 749.3 m.m. (Bunsen), the amount of ammonia found in the contents of the burette below this point would, of course, go on increasing indefinitely, depending simply on the length of time allowed for condensation and the more or less free supply of the gas. It appears that ammonium hydroxide, if such a substance exist, or the solution of ammonia in water in proportion corresponding to such a compound, continues to dissolve gaseous ammonia, or mixes with liquefied ammonia, down to and beyond the normal boiling-point of the latter, but the proportion dissolved is much greater than would be called for by an extension of the curve representing solubility at temperatures above 0° .

As has been stated, measurement of the volumes of the samples taken could not be very satisfactorily carried out. It may be mentioned, however, that a comparison of volume with weight gave for the liquid at -30° a density of about 0.718, and for that at -40° about 0.731, as referred to water at $+4^{\circ}$. The latter figure being greater than the former may probably be accounted for by the very high value of the coefficient of dilatation for liquefied gases. Calculating from Jolly's results, the density of liquefied ammonia itself at -40° ought to be something like 0.673.—*American Chemical Journal*, xix., No. 9.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING NOVEMBER 30TH, 1897.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolitan Water Act, 1871.

London, December 10th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 182 samples of water collected by us during the past month, at the several places and on the several days indicated, from the

mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Nov. 1st to Nov. 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 182 samples examined by us, one was recorded as "clear but dull," the remainder being clear, bright, and well filtered.

The rainfall at Oxford shows a further serious deficiency, the actual fall during the month being only 1.15 inches, against a thirty years' average of 2.10 inches, making a deficit of 0.95 inch. The total deficit for the year is now 1.36 inches.

Our bacteriological examinations of 252 samples have given the results recorded in the following table; we have also examined 77 other samples, from special wells, stand-pipes, &c., making a total of 329 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 26 samples) ..	488
New River, filtered (mean of 25 samples) ..	13
Thames, unfiltered (mean of 26 samples) ..	27,173
Thames water, from the clear water wells of five Thames-derived supplies (mean of 122 samples)	27
Ditto ditto highest	218
Ditto ditto lowest	1
River Lea, unfiltered (mean of 26 samples) ..	593
River Lea, from the clear water well of the East London Water Company (mean of 26 samples)	12

The very small rainfall during the past three months has had the usual good effect on the quality of the London water supply, which is now both chemically and bacteriologically excellent.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.
JAMES DEWAR.

A REVISION OF THE ATOMIC WEIGHT OF NICKEL.*

FIRST PAPER.—THE ANALYSIS OF NICKELOUS BROMIDE.

By THEODORE WILLIAM RICHARDS
and
ALLERTON SEWARD CUSHMAN.

(Concluded from p. 296).

WHEN the manipulation had thus been mastered, the art of preparing absolutely pure nickelous bromide had been perfected (see Analysis 7), and the atomic weight of nickel had been approximately determined, the method of procedure in subsequent analyses was changed. The now perfectly clear solution was treated with just enough argentic nitrate, prepared from the purest weighed silver, to complete the precipitation. The mean between the two possible end-points was determined by titrating back.

* Contribution from the Chemical Laboratory of Harvard College, from the *Proceedings of the American Academy of Arts and Sciences*, vol. xxxiii., No. 7.

wards and forwards with hundredth normal argentic nitrate and hydrobromic acid solutions (for the details see *Proc. Amer. Acad.*, xxx., 384); and thus was determined the ratio of silver to nickelous bromide entitled Series III. After this end-point had been determined, a slight excess of argentic nitrate was added to the solution, and the whole was violently shaken. The precipitate was collected upon a Gooch crucible, washed with water containing a trace of argentic nitrate, later with pure water, and finally dried and weighed. The traces of asbestos carried away by the wash water were of course determined, and all the usual precautions were taken to insure great accuracy. Thus was obtained the series of results given in Series II.

The Atomic Weight of Nickel.

O = 16.000; Ag = 107.93.

FIRST SERIES (PRELIMINARY).—RATIO = 2AgBr : NiBr₂.

Number of expt.	Sample of NiBr ₂ .	Weight of nickelous bromide in vacuum. Grms.	Weight of insoluble residue. M. grms.	Weight of argentic bromide in vacuum. Grms.	Atomic weight of nickel.
1.	I.	2.26113	3.22	3.88769	58.646
2.	I.	2.80668	7.08	4.82431	58.708
3.	II.	1.41317	3.05	2.42880	58.716
4.	II.	1.71759	0.88	2.95307	58.650
5.	III.	2.48565	5.24	4.27357	58.651
6.	III.	4.32997	15.83	7.44280	58.700
7.	III.	2.18072	0.00	3.74856	58.693

Average.. .. 58.680

SECOND SERIES.—RATIO = 2AgBr : NiBr₂.

Number of expt.	Sample of NiBr ₂ .	Weight of nickelous bromide in vacuum. Grms.	Weight of argentic bromide in vacuum. Grms.	Atomic weight of nickel.
8.	III.	3.28039	5.63892	58.691
9.	III.	2.70044	4.64208	58.686
10.	III.	3.38230	5.81391	58.698
11.	III.	1.33459	2.29435	58.670
12.	IV.	1.25054	2.14963	58.693
13.	IV.	1.32278	2.27384	58.690
14.	IV.	2.24452	3.85805	58.705

Average.. .. 58.690

THIRD SERIES.—RATIO = 2AgBr : NiBr₂.

Number of expt.	Sample of NiBr ₂ .	Weight of nickelous bromide in vacuum. Grms.	Weight of silver in vacuum. Grms.	Atomic weight of nickel.
8.	III.	3.28039	3.23910	58.701
9.	III.	2.70044	2.66036	58.709
10.	III.	3.38230	3.33090	58.689
11.	III.	1.33459	1.31787	58.689
12.	IV.	1.25054	1.23482	58.698
13.	IV.	1.32278	1.30629	58.675
14.	IV.	2.24452	2.21652	58.676

Average.. .. 58.691

In the table above, the first column records the number of the experiment, the second records the number of the sample of nickelous bromide used, while the third records the weight of this salt taken. The extreme right hand column contains the atomic weight of nickel computed from the values contained in the one just to the left of it, and those contained in the third.

A very interesting evidence of the accuracy of these results is the relationship between the amount of silver taken and the amount of argentic bromide obtained. From the second and third series, we find that 15.51556 grms. of nickelous bromide yielded 26.67078 grms. of argentic bromide, requiring 15.32086 grms. of silver. This leads

to the inference that argentic bromide contains 57.444 per cent of silver—a quantity which agrees essentially with the value 57.445 per cent found by Stas. Since the bromine used had been already found to be free from other halogens, and the silver was known to be perfectly pure, we have in these results conclusive proof that no nickel salt was occluded by the argentic bromide, as well as a satisfactory "check" upon the accuracy of the work.

When we examine the results with respect to the various samples of the salt analysed, we find a very interesting and satisfying uniformity. The four samples of nickelous bromide gave the following results for the atomic weight of nickel:—

Sample I...	58.677
" II...	58.683
" III...	58.688
" IV...	58.689

The slight rise in the value with increasing purity is not large enough to have any weight, for there are analyses in the lowest series giving higher individual results than any in the highest series. Hence we are forced to the conclusion that the least carefully purified specimens of nickelous bromide must have been essentially identical with the most carefully purified. The chances are evidently exceedingly small that the impurities would so combine as exactly to counterbalance one another.

The further discussion of this important question will be reserved until more experimental work has been done. For the present, it is our opinion, at this first halting place in a long investigation, that the atomic weight of nickel cannot be far from 58.69 if O = 16.00, or 58.25 if O = 15.88.

ON THE CHLORONITRIDES OF PHOSPHORUS.*

By H. N. STOKES.

In a former article (*Am. Chem. Journ.*, xvii., 275, 1895; *Ber. d. Chem. Ges.*, xxviii., 437) I have shown that in addition to the phosphonitric chloride, $P_2N_2Cl_6$, discovered by Liebig, there exists another, $P_4N_4Cl_8$, of similar properties, which is formed at the same time, but in smaller quantity. The opinion was expressed that these bodies belong to a series of polymers, $(PNCl_2)_n$, the existence of other members of which was indicated by the formation, in small amount, of a liquid of the same empirical composition (*Am. Chem. Journ.*, xvii., 277, 280, 290). The yield of this secondary product—only 2 per cent of the theoretical or 1 per cent of the pentachloride used—was too small to allow of its preparation in quantities large enough to admit of the isolation of its supposed constituents, but a fractional distillation of the few grms. at my disposal showed that it contained crystalline substances of higher boiling-points than those of the two bodies thus far known.

The method of preparation then employed consisted in distilling phosphorus pentachloride with a large excess of ammonium chloride in a retort, at atmospheric pressure; it offered but little prospect of obtaining the higher members. The total yield of phosphonitric chloride was but 15 per cent of the theoretical, most of the pentachloride being converted into "phospham" by the excess of ammonium chloride, while only those members could be obtained which distil unchanged at atmospheric pressure. Decreasing the amount of ammonium chloride resulted only in a loss of pentachloride by volatilisation, without increasing the yield of the bodies sought after.

* Published by permission of the Director of the United States Geological Survey. From the *American Chemical Journal*, vol. xix., No. 9, November, 1897.

† I propose in future to use the term *phosphorus chloronitride* to denote any body composed of phosphorus, nitrogen, and chlorine, the name *phosphonitric chloride* being reserved for chloronitrides belonging to the series $(PNCl_2)_n$.

The following method has been found to give entirely satisfactory results; several new bodies have been obtained, and the simpler phosphonitric chlorides at least are now easily accessible substances:—If equal molecular weights of phosphorus pentachloride and ammonium chloride be heated in a sealed tube, there results a mixture of chloronitrides, which is partly crystalline and soluble in gasoline, but for the greater part liquid and insoluble in this solvent, and of a high degree of complexity. This may be distilled almost without residue, the distillate being a crystalline mass, impregnated with an oil, and composed almost wholly of a mixture of members of the series $(\text{PNCl}_2)_n$ in nearly theoretical amount, containing about 50 per cent $\text{P}_3\text{N}_3\text{Cl}_6$, and 25 per cent $\text{P}_4\text{N}_4\text{Cl}_8$, the remainder consisting of the higher homologues. From this distillate the new bodies, with one exception, have been isolated.

The series, as at present known, consists of the following (the melting- and boiling-points are corrected):—

	Melting-point.	Boiling-point.	
		13 m.m.	760 m.m.
Triphosphonitric chloride, $(\text{PNCl}_2)_3$	114°	127°	256.5°*
Tetraphosphonitric chloride, $(\text{PNCl}_2)_4$	123.5°	188°	328.5°†
Pentaphosphonitric chloride, $(\text{PNCl}_2)_5$	40.5-41°	223-224.3°	Polymerises
Hexaphosphonitric chloride, $(\text{PNCl}_2)_6$	91°	261-263°	Polymerises
Heptaphosphonitric chloride, $(\text{PNCl}_2)_7$.			
Liquid at	-18°	289-294°	Polymerises

Polyphosphonitric chloride, $(\text{PNCl}_2)_x$ Below red heat. Depolymerises on distillation.

* 183.8° at 100 m.m.

† 242° at 100 m.m.

There were obtained, further, a liquid residue of the same empirical composition, of a mean molecular weight corresponding nearly to $(\text{PNCl}_2)_{12}$, and a small amount of a chloronitride, $\text{P}_6\text{N}_6\text{Cl}_{12}$, not belonging to the above series. The absence of the lower members, PNCl_2 and $(\text{PNCl}_2)_2$, is remarkable, and theoretically significant. Indications of a trace of a substance more volatile than the compound $(\text{PNCl}_2)_3$ and of similar but stronger odour, were observed, but there is no evidence that it consists of one of the missing bodies.

One of the most remarkable properties of the phosphonitric chlorides is that each member of the series is converted by heat into the rubber-like *polyphosphonitric chloride*, a body, or mixture of bodies, of very high molecular weight, which is highly elastic and insoluble in all neutral solvents, but which swells enormously in benzene, and which, on distilling at a higher temperature, breaks down into a mixture of all the lower members mentioned above, which can then be separated by appropriate means. In this way it is possible to convert any phosphonitric chloride quantitatively into any other by heat and distillation alone. In preparing any desired member, therefore, we are not limited to the quantity obtained from the first reaction product, but may work the residues over and over again until completely converted into the body sought after. With the exception of a few cases, in which the number of members is limited, as the aldehydes and cyanic acids, this series is therefore unique; I know of no other series of inorganic compounds in which this is possible. Polymerisation takes place slowly, but perceptibly, at 250°, and is almost instantaneous at 350°, while depolymerisation begins at about 350°, and is rapid at a temperature close to incipient red heat. Triphosphonitric chloride, $\text{P}_3\text{N}_3\text{Cl}_6$, is the only member which can be distilled in considerable amount at atmospheric pressure without considerable polymerisation, and even this polymerises almost completely on long boiling; at 760

m.m. pressure the tetra-compound, $\text{P}_4\text{N}_4\text{Cl}_8$, boils at 328.5°, a temperature at which polymerisation occurs quite rapidly, but this, as well as the penta-compound, $\text{P}_5\text{N}_5\text{Cl}_{10}$, and the hexa-compound, $\text{P}_6\text{N}_6\text{Cl}_{12}$, can readily be distilled at 13 m.m.; the hepta-compound, $\text{P}_7\text{N}_7\text{Cl}_{14}$, suffers marked polymerisation on distilling even at this pressure, and its isolation is therefore attended with much loss. Owing to the rapid change at higher temperatures, I have been unable to isolate any of the higher members, which remain as a considerable oily residuum, and there seems to be but little probability of this being effected by any known method, unless by distilling in a nearly absolute vacuum.

The greatest difficulty in the separation of the members is caused by polymerisation. It requires but a small amount of polyphosphonitric chloride to cause the liquid to thicken or gelatinise, and therefore to be incapable of further distillation; and some of this body is always formed in the course of a prolonged fractioning of the higher members. It was found, however, that this polymer is much more easily attacked by water than the lower members; when signs of polymerisation are observed, it is only necessary to interrupt the distillation and heat the residue for some time with water, when the resulting oil is again in a condition to continue fractioning.

The loss in this operation is small, but the tediousness of a fractional distillation is thereby extraordinarily increased.

It is noteworthy that no regular progression exists in the melting-points of the phosphonitric chlorides, and the same is true of their solubility in the ordinary neutral solvents, but the solubility varies in the same sense as the fusibility. Of the members of known molecular weight, the second, tetraphosphonitric chloride, is the least soluble and has the highest melting-point, while the corresponding tetrametaphosphoric acid is the least soluble and most stable of the derived acids. With respect to their stability towards water, the new members (polyphosphonitric chloride excepted) resemble those already described, being scarcely attacked by prolonged boiling. In ethereal solution, however, there is a perceptible decrease of stability towards water as we rise in the series—a fact already noted with regard to the first two members (*Am. Chem. Journ.*, xvii., 289).

Notwithstanding the high molecular weight of the bodies isolated, no indication of isomers has been observed, although the fractioning was carried out very thoroughly up to 300° at 13 m.m.

The investigation will be continued with the object of obtaining the metaphosphoric acids, and, if possible, the two missing lowest members of the phosphonitric chloride series. The right of further investigation in this field, however, is not reserved.

Experimental Part.

A mixture (which need not be very intimate) of 4 parts perfectly dry phosphorus pentachloride and 1 part ammonium chloride, as required by the equation $\text{PCl}_5 + \text{NH}_4\text{Cl} = \text{PNCl}_2 + 4\text{HCl}$, is introduced into an ordinary "bomb" tube, which has previously been drawn out to a neck. It is practicable to fill the tube entirely to the neck, so that the charge for a tube of ordinary dimensions is about 125 grms., yielding 50–55 grms. of chloronitrides. After sealing, the length of the neck, exclusive of the rather long capillary, should be about 10 c.m. As the mixture liberates 55 per cent hydrochloric acid, it is necessary to regulate the heating with great care and to open the tube repeatedly. The temperature of the furnace is allowed to rise to 150°, at which the reaction begins, when the gas is at once shut off, and the tube opened at about 100° (in the furnace!). This operation is repeated several times, the temperature being allowed to rise 1–2° higher each time. When the evolution of hydrochloric acid has slackened and the contents of the tube are mainly liquid while hot, the temperature may be carried to 200° or higher, until little

or no gas is given off. The operation requires care and judgment, but with careful working it is possible to avoid explosions, and to obtain with a four-tube furnace about 200 grms. of mixed chloronitrides in sixteen hours.

The contents of the tube, after cooling, generally consist of a buttery mass or of a thick yellow liquid filled with fine prisms and plates; if heated much above 200°, the liquid frequently separates into two layers. The crystals are soluble in gasoline, but the bulk of the product remains as an immiscible oil.

The neck of the tube is now bent down, the tube placed in an inclined combustion furnace, and by cautious heating, finally to incipient redness, the contents are distilled out. There remains in the tube a very voluminous spongy black residue, of inconsiderable weight, due to unavoidable impurities, and to the impossibility of causing complete reaction in the sense of the above equation. The distillate consists of a crystalline mass impregnated with a yellow oil, and contains about 95 per cent of the theoretical amount of phosphonitrilic chlorides, with some phosphorus pentachloride, the chloronitride $P_6N_7Cl_9$, and other substances of unknown nature. Before proceeding further, it is necessary to remove the pentachloride, and for this purpose the distillate is melted, poured into cold water, and the flask heated in the water-bath for about two hours, the liquids being mixed by blowing air through them. The chloronitrides are then allowed to clear under the hot water, and forced out by means of a wash-bottle arrangement; a separatory funnel cannot be used, as the substance solidifies in the neck, and if allowed to solidify under the wash water it absorbs so much of this as to cause annoyance in the subsequent distillation. Special drying before distilling is unnecessary.

The product is then distilled up to 200° at 13 to 15 m.m., using an Anschütz flask, as the distillate solidifies instantly on cooling. The residue, containing the members $P_5N_5Cl_{10}$ up, is set aside for later systematic fractional distillation.

(This residue contains the small amount of $P_6N_7Cl_9$, formed as a secondary product of the original reagents, and as this is apt to cause inconvenience at a later stage, by accumulating with the $P_6N_6Cl_{12}$, it is perhaps well to remove as much as possible at this point. For this purpose the residue is allowed to stand for a day or two at the room temperature, and the crystals removed by sucking out under a good vacuum, beat in a large Gooch crucible. The filtrate is cooled for a day or two in a refrigerator, and the new crop of crystals separated in the same way, the filtering flask being allowed to stand in the ice-box. The oily filtrate is set aside, and the united crystalline products distilled up to 240° at 13 m.m., whereby most of the $P_5N_5Cl_{10}$ passes over. The residue, consisting of $P_6N_6Cl_{12}$, the small amount of $P_6N_7Cl_9$, and the adhering oil, is allowed to crystallise in the refrigerator, and the viscous mass is extracted several times with small amounts of gasoline (boiling at 50° to 80°). The residue is boiled with benzene, which extracts the $P_6N_7Cl_9$, which crystallises on concentrating and cooling. The portion dissolved by the gasoline is worked up with the other residues. This is the method actually employed, but I am not entirely convinced of its necessity, as it is not possible to remove all the $P_6N_7Cl_9$ in this way).

The distillate, about 70 per cent, consists essentially of $P_3N_3Cl_6$ and $P_4N_4Cl_8$, which, if desired, may be easily separated by fractional distillation *in vacuo*, followed by crystallisation from benzene. This is more convenient than the method of separating by steam (*Amer. Chem. Journ.*, xvii., 280). If it is desired to convert it into the higher members, it is placed in a combustion tube bent down at about 20 c.m. from the open end, and which it should not fill more than one-half after melting. This is laid in an inclined combustion furnace, and heated to gentle boiling of the contents. It is well to heat the tube somewhat strongly at a short distance above the liquid, as

superheating the vapour promotes polymerisation. The time required for polymerisation varies greatly; pure triphosphonitrilic chloride may require two hours or more; with the above mixture the time is less, and is shorter the higher the boiling-point; it is shortened by adding already gelatinised substance, which causes the liquid to thicken, and may then be but a few minutes; it is also shortened by heating under pressure. Sooner or later the liquid begins to thicken, and finally it is converted into a stiff, transparent mass, with little or no liquid, and generally discoloured by traces of organic matter. The tube is then connected with a long-necked receiver, exhausted, and the depolymerisation and distillation effected by heating, from the front backward, to incipient redness. This part of the operation proceeds rapidly, as it is only necessary to guard against frothing over, and to ensure complete condensation, the latter being easily effected by having the limb of the tube at least 20 c.m. long: 100 grms. can be worked up at one time, and the tube can be used repeatedly. The residue does not weigh more than a few m.grms. The distillation may also be made at atmospheric pressure, but the yield of higher products is thereby diminished. The distillate, which entirely resembles that first obtained, except in containing no phosphorus pentachloride and no $P_6N_7Cl_9$, is distilled as before, the washing being omitted. In this way the whole quantity of material can finally be converted into a mixture of members higher than $P_4N_4Cl_8$.

The united residues boiling above 200° are now submitted to systematic fractional distillation at 13 to 15 m.m., using an Anschütz flask, provided with a "trap," to prevent flowing back. During the first distillation polymerisation generally begins when the temperature of the bath has reached 270°, but with later distillations at a higher temperature, and the higher the purer the fractions are. When polymerisation begins, which is indicated by frothing and thickening, the operation is interrupted, and the residue heated in the flask with water in the water-bath until it has completely liquefied, which is assisted by agitation, the oil separated,* and the distillation continued. It has not been found practicable to continue the distillation at a higher temperature than that obtained by heating the bath to 370°, for the liquid begins to polymerise in a few moments, and but an inconsiderable distillate can be obtained. Moreover, at this temperature the polymer shows signs of breaking down into simpler bodies, and the distillate does not consist only of high-boiling members. The total amount of final residue is not very great, and as shown below consists likewise of phosphonitrilic chlorides of still higher molecular weight. In later distillations, from 200° upward, polymerisation usually stops the process at 260° to 270°, but after appropriate washing the residue may be distilled to a much higher temperature. After 8 to 10 distillations three main fractions are obtained, which are then worked up separately. As $P_5N_5Cl_{10}$, though crystalline, is extremely soluble, it is necessary to carry out the distillations with the first main fraction until a practically sharp boiling-point is obtained, in which connection it may be noted that at 17 to 20 m.m. a change of 1 m.m. pressure causes a change of about 1° in the boiling-point, and at 13 m.m. a change of about 2°. $P_7N_7Cl_{14}$, being liquid, must also be isolated by distillation only, but at its boiling-point polymerisation is so rapid that great loss ensues during a series of distillations. The final purification of $P_6N_6Cl_{12}$ can be effected by repeated re-crystallisation from benzene, combined with treatment with gasoline to remove the $P_6N_7Cl_9$, which always accompanies it, having nearly the same boiling-point: a complete separation of these two bodies can scarcely be effected by distillation alone.

Owing to many modifications introduced in developing the above method, no accurate statement of the yield can be given; the final product was about 225 grms.

* In this case a separatory funnel may be used, as the higher chloronitrides are liquid below 80°.

$P_3N_3Cl_{10}$, 110 grms. $P_6N_6Cl_{12}$, 10 grms. $P_7N_7Cl_{14}$, and 5 grms. $P_5N_5Cl_8$.

Analytical Methods.

With the exception of polyphosphonitrilic chloride, the chloronitrides were analysed by decomposing in the following manner:—

For *phosphorus*, by warming with alcohol and a little ammonia in a platinum crucible until completely dissolved, evaporating to dryness, and heating to fuming for an hour with strong sulphuric acid, the crucible being kept covered.

For *nitrogen*, by treating as above, omitting the ammonia.

For *chlorine*, by heating with alcohol and ammonia. It is necessary to precipitate with silver nitrate in the presence of a large volume of 10 per cent nitric acid and to filter hot, in order to avoid the formation of silver metaphosphimates, which are difficultly soluble in dilute nitric acid.

In decomposing polyphosphonitrilic chloride, which is attacked by water alone, the alcohol was omitted. The method of Carius was used for determining chlorine, as it was found that otherwise compounds insoluble in dilute nitric acid were formed. For the other chloronitrides this method offers no advantage.

Molecular weight determinations were made by the boiling-point method with the apparatus of Hite (*Amer. Chem. Journ.*, xvii., 512), using as solvent carefully purified and dried benzene.

(The molecular weight of $P_3N_3Cl_6$ has been determined by the vapour density method (*J. Chem. Soc.*, [2], ii., 225; *Amer. Chem. Journ.*, xvii., 283). A series of determinations by the boiling-point method gave 346, 350, 353. Calculated, 347.9).

Pentaphosphonitrilic chloride, $P_5N_5Cl_{10}$.—This body, carefully purified by fractional distillation, as described above, gave on analysis:—

	Calculated for $P_5N_5Cl_{10}$	Found.
P	26.75	26.87
N	12.11	12.05
Cl	61.14	61.42

Ratio, P : N : Cl = 1 : 0.99 : 2.00.

Molecular Weight. Solvent: Benzene.

Grms. solvent.	Grms. substance.	Elevation.	Molecular weight found.	Percentage variation from theoretical.
46.49	1.4688	0.137°	619	+6.2
"	2.9439	0.287°	589	+1.6
"	4.4337	0.437°	583	+0.5

Calculated for $P_5N_5Cl_{10}$, 579.8.

Pentaphosphonitrilic chloride fuses at 40.5°—41°, and boils at 223°—224.3° (corr.) at 13 m.m. Its vapour is without the pronounced and characteristic aromatic odour possessed by that of triphosphonitrilic chloride. At its melting-point it is miscible in all proportions with benzene, gasoline, ether, and carbon disulphide, and cannot be re-crystallised from any of these solvents, in fact, small fragments liquefy instantly in their concentrated vapours. Glacial acetic acid also dissolves it quite readily, and from this solution water throws it out as an oil, solidifying at once on touching. It shows a decided tendency to superfusion, especially when not absolutely pure; when left by evaporating its ether or benzene solution, it may remain liquid for days, but solidifies at once on touching with a glass rod, usually to a decidedly crystalline mass, at other times to a transparent glass. The pure substance, when fused, slowly solidifies, long flat crystals shooting out through the liquid, which are limited only by the size of the vessel, crystals of 10 c.m. in length being readily obtained. It contracts greatly on solidifying. When pure the solidified mass is naturally dry, but the least contamination with other members of the series causes a portion to remain liquid, which is easily detected by crushing on a piece of filter-paper; this is a very good

test of its purity. This tendency to superfusion must be borne in mind in separating it by fractional distillation; a nearly pure sample will remain liquid much longer than the higher or lower fractions. In ether solution it is perceptibly more easily attacked by water than the preceding chloronitride, but hot water alone is almost without action.

Hexaphosphonitrilic Chloride, $P_6N_6Cl_{12}$.—After repeated crystallisation from benzene, this gave—

	Calculated for $P_6N_6Cl_{12}$	Found.
P	26.75	26.98
N	12.11	12.37
Cl	61.14	60.98

Ratio, P : N : Cl = 1 : 1.01 : 1.98.

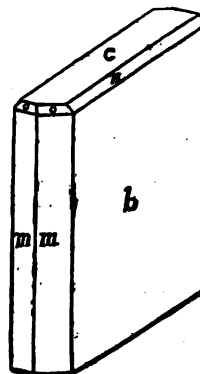
Molecular Weight. Solvent: Benzene.

Grms. solvent.	Grms. substance.	Elevation.	Molecular weight.	Percentage variation from theoretical.
47.12	1.8058	0.152°	673	-3.2
"	3.6190	0.293°	700	+0.6
45.72	1.0101	0.083°	711	+2.1
"	4.4898	0.365°	718	+3.2
"	8.0070	0.664°	704	+1.2

Calculated for $P_6N_6Cl_{12}$, 695.8.

Hexaphosphonitrilic chloride fuses at 91° (corr.), and boils at 261°—263° (corr.) at 13 m.m., and at 281°—282° (corr.) at 26 m.m. It may be re-crystallised from benzene, in which, however, it is more soluble than triphosphonitrilic chloride; ether, gasoline, and carbon disulphide also dissolve it readily; in alcohol it dissolves somewhat slowly with decomposition. It shows no tendency to superfusion. It crystallises well, in rather large crystals, which were examined by Mr. Wirt Tassin, to whom I am indebted for the following statement:—

" $P_6N_6Cl_{12}$ crystallises in the orthorhombic system in long prismatic crystals, showing the following forms:—*c* (001), *b* (010), *m* (110), *o* (111), *n* (011). Of these *c* is the dominant form; *b* large and well developed; *m* fair, though usually narrow; and *o* and *n* small and usually in



similar development. *c*, *b*, *m*, *o* is the combination occurring most frequently; less often *c*, *b*, *m*, *n*, *o*; and rarely *c*, *b*, *m*. Angles *m* : *m* 57° 28', *b* : *m* 61° 16', *b* : *n* 40° 23', *n* : *c* 49° 37'. Axial ratio *a* : *b* : *c* = 0.54824 : 1 : 1.17568. The crystals are optically positive. Plane of the optic axes (100); colourless to white; transparent; and have a perfect basal cleavage."

It is scarcely attacked by boiling water, but if kept in moist air it very slowly evolves hydrochloric acid. Its ether solution, shaken with water, slowly gives a metaphosphimic acid; syrupy chlorhydrines are formed as intermediate products.

(To be continued).

* A contamination with $P_3N_3Cl_6$ may be detected by treatment with gasoline, when the much smaller crystals of the latter are seen to dissolve much more slowly.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, December 2nd, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

CERTIFICATES were read for the first time in favour of Messrs. Charles Edward Britain, B.Sc., 11, Highfield, Scarborough; William Arthur Caldecott, B.A., Box 1891, Johannesburg, S.A.R.; John Cooper, B.Sc., 20, Derwent-water Road, Gateshead; Frederick Cowling, Clay Cross, near Chesterfield; Wilbraham T. A. Edwards, Reduit, Mauritius; Frederick Gilderdale, 3, Havelock Street, Newcastle; William Setten Gilles, Coniston, Cedars Road, Beckenham; William Hobson Mills, B.A., Jesus College, Cambridge; Frank Forster Renwick, Glengall, Woodford Green, Essex; William Colebrook Reynolds, 64, Lydford Road, Paddington, W.; Andrew Jamieson Walker, B.A., Killycadden, Killygordon, Co. Donegal; Ernest Charles Weismüller, 30, Pepys Road S., New Cross, S.E.

The following were duly elected Fellows of the Society:—John Ball, Ph.D.; William Ball; Alec. Alfred Beadle; Richard Oxley Burland, J.P.; Alexander McLean Cameron; Alexander Clarkson; Frank Collingridge, B.Sc.; James Murray Crofts, B.A.; John Daniell; Andrew James Dixon, F.I.C.; Oscar Guttman, F.I.C.; Robert Hamilton; John Harger, B.Sc., Ph.D.; James Walter Horseman; Charles Kelly; Tom Lemmey, B.A.; James Scott MacLaurin, D.Sc.; Alan Macmullen, B.A.; Charles Jodrell Mansford, B.A.; Edward Masters; John A. Mathews, M.A. M.Sc.; Philip George Gregory Moon; James Charles Philip, B.Sc., Ph.D.; Alexander Ferguson Reid; Ernest Henry Roberts; Edward Sydney Simpson, B.E.; Robert Francis Wood Smith; Thomas Southern, Jun.; Frederick William Steel; Michael Edmund Stephens; George Stubbs; Edward Howard Tripp, Ph.D.; John Scriven Turner; Framjee Khursedjee Viccajee; Percy John Vinter, M.A.; Arthur James While; Francis Samuel Young, M.A.

The PRESIDENT called the attention of Fellows to the fact that, although according to the Council regulations no paper could be announced which had not been received, it was open to any Fellow to make a communication at a meeting of the Society in the event of there being available time, provided that he handed a written statement to the Secretaries of the essence of his communication.

In answer to a question from Dr. Hake, the PRESIDENT said that the appearance of such communications in the *Proceedings* would be subject to the editorial discretion of the Secretaries, and that when presented as full papers they would come before the Publication Committee in the usual way.

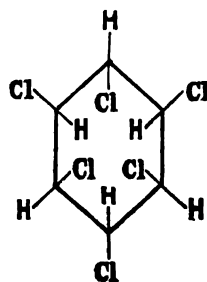
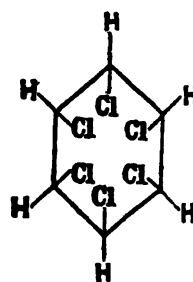
The following papers were read:—

*124. "The Representation of the Isomeric Benzene Hexachlorides by Collie's Space-formula." By FRANCIS EDWARD MATTHEWS, Ph.D.

This paper discusses Collie's space-formula for benzene from the point of view of the halogen hex-addition compounds. The formula is shown to explain the existence of two isomeric hexachlorides satisfactorily, and the accompanying formulae are proposed for these compounds.

These formulae likewise explain the differences in stability towards alcoholic alkalis. The α -substance, containing the chlorine in the ortho-position to, and on the same side of the carbon nucleus as the hydrogen atoms with which it is removed in the form of hydrogen chloride, is readily decomposed; the β -substance, in which the hydrogen and chlorine are on opposite sides of the nucleus, has much greater stability. The remainder of the paper discusses the formation of benzene di-derivatives from mono-derivatives, and it is maintained that their

formation is best explained by assuming the previous formation of unstable *ortho*-addition compounds instead of unstable *meta*-compounds, as Collie has suggested.

 α -Compound. β -Compound.

DISCUSSION.

Dr. WYNNE asked whether any explanation could be given of the formation of 1 : 2 : 4-trichlorobenzene from the benzene hexachlorides by the action of alcoholic potash. The usually accepted formulae for benzene gave no clue to the reason for the production of unsymmetrical derivatives in such cases, and he was unable to see that the formulae now proposed were more satisfactory in this respect.

Dr. LAPWORTH pointed out that the author's statement as to the impossibility of explaining the production of two different hexachlorides from benzene by means of any formula prior to Collie's, appeared to need qualification. It is easily seen that Kekulé's formula represents several stereoisomeric substances, as each "doubly bound" pair of carbon atoms may form a fumaroid or maleoid combination. Taking into consideration the circumstance that, for a number of reasons, the formula must be considered as a labile one, the positions of the ethylenic and single linkings being supposed to alternate, it is not impossible that, under the conditions of interaction of chlorine and benzene, the latter may react in the form of two or more of its possible stereoisomers. The addition of chlorine to a pair of carbon atoms double bound as in fumaroid compounds would, of course, afford a trans-dichloro-derivative, whilst a cis-dichloro-compound would result if the original combination were of the maleoid type. Kekulé's formula, therefore, would appear to afford a perfectly satisfactory explanation of the production of more than one benzene hexachloride.

Mr. E. J. PARRY asked if Dr. Matthews had any experimental evidence for assigning the symmetrical formula to the α -hexachloride, and the unsymmetrical to the β compound. If the explanations of substitution offered by Collie and the author were correct, there should certainly be twice as much ortho- as para-dichlorobenzene produced when chlorobenzene is chlorinated. Further, he could not understand why, if these explanations were correct, the nitration of chlorobenzene should give ortho- and para-compounds whilst the chlorination of nitrobenzene, or the nitration of nitrobenzene, should yield meta-compounds.

Dr. MATTHEWS, in reply, stated that it had always seemed to him a remarkable fact that 1 : 2 : 4-trichlorobenzene alone was produced by the action of alkalis upon the benzene hexachlorides. He had made several attempts with large quantities of material to isolate the symmetrical modification, but always without result. The formation of the 1 : 2 : 4-compound could, however, be easily explained, either by Kekulé's or Collie's formula.

With regard to Dr. Lapworth's remarks, the formula proposed above account for the production of two and not of a greater number of isomerides which might be expected if these hexachlorides were regarded as cis- and trans-modifications; whilst certain properties of the compounds do not seem to harmonise with the idea that they are stereoisomerides.

The answer to Mr. Parry's questions are contained in the paper itself; the difference in stability of the two hexachlorides towards alkalis is explained by assuming that hydrogen chloride is more readily removed from atoms in the ortho-position and on the same side of the carbon nucleus than from those in which these conditions do not obtain. Hence the above formulæ were assigned.

*125. "Compounds of Piperidine with Phenols." By OTTO ROSENHEIM, Ph.D., and PHILIP SCHIDROWITZ, Ph.D.

With a view of obtaining substances of the general formula $(C_6H_{4-n})(C_5H_{10}N)_n$, which seemed to be of interest on account of their relation to the phenylenediamines and polyamines, the authors studied the action of piperidine on phenols and their derivatives in the presence of dehydrating agents. Although so far unsuccessful in this direction, a series of addition products in the nature of salts was observed, in which piperidine acts as the base and the phenol as the acid. They are well crystallised compounds, easily obtained by the interaction of their components, usually in ethereal solution. They are resolved into their constituents by strong acid or alkalis. M. Oechser de Coninck (C. R., 1897, cxxiv., 563) describes a number of colour reactions obtained by the action of piperidine and other bases on phenols in dilute aqueous solution, but has apparently not observed the formation of the addition compounds described in the paper.

The influence of the number and position of the oxy- and nitro-groups in the phenols on the additive capacity of the piperidine molecule was studied, but no general rule could be deduced.

The following compounds were analysed, and are described in the paper:—Compounds of piperidine (1 mol.) with pyrocatechol (2 mols.), guaiacol (2 mols.), hydroquinone (1 mol.), pyrogallol (1 mol.), vanillin (1 mol.), o- and p-nitrophenol (1 mol.), picric acid (1 mol.), 1:2:4-dinitronaphthol (1 mol.). Phenol, p-chlorophenol, resorcinol, phloroglucinol, m-nitrophenol, and α- and β-naphthol did not furnish crystalline compounds.

EDINBURGH UNIVERSITY CHEMICAL SOCIETY.

Monday, November 29th, 1897.

Mr. W. W. TAYLOR, M.A., B.Sc., in the Chair.

DR. BOLAM read a paper on "Electrolysis in Organic Chemistry."

The paper was mainly historical, and dealt first with the work of Kolbe, Bourgoin, and Kekulé. It was pointed out that Löb had recently given a confirmation of Kekulé's "anhydride theory" by electrolysis of phthalic acid.

When phthalic acid is dissolved in alcohol and a few drops of an acid added to increase the conductivity, the passage of a weak current through the solution for some time gives an almost quantitative yield of phthalic anhydride. Longer duration of the current results in the formation of phthalic ether in large quantity.

Dr. Bolam also referred to the use of the alternate current, and concluded by referring to some technical applications in organic chemistry.

There was a large attendance, and Dr. Bolam was thanked for his paper.

CORRESPONDENCE.

ZINC IN WATER.

To the Editor of the Chemical News.

SIR,—I read in the CHEMICAL NEWS (vol. lxxvi., p. 293) that Mr. Percy Richards has detected zinc in a sample of

water used for drinking purposes, in Berkshire, which was supplied by a galvanised iron pipe.

It may be interesting to note that among the samples of water examined last year in my laboratory there were some from the island of Madeira which presented the same character, and which had, likewise, been conducted into the town of Funchal by galvanised iron pipes. There was a large deposit of oxide and carbonate of zinc in one of the bottles.

Putting aside the zinc contamination, the water was of great purity. As zinc is a metal whose compounds have a noxious action upon the economy, it is evident that galvanised iron pipe cannot be used with safety to supply water for drinking.—I am, &c.,

T. L. PHIPSON, Ph.D.

Cass Mia, Putney, S.W.,
December 18, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xvii.-xviii., Nos. 16-17.

Action of Chlorine on Pentachlorethane in the presence of Chloride of Aluminium.—A. Mouneyrat.—Although chloride of aluminium has a special action on chloral (removing oxygen), it seemed to the author to be of interest to verify whether or not this powerful synthetical agent might not be a chloridising agent in the acyclic series. He therefore placed 150 grms. of pentachlorethane (C_2Cl_5H) in a flask heated to 70° and provided with a vertical condenser, and exposed to diffused light. Dry chlorine was passed through for thirty hours. This gas was not absorbed, no hydrochloric acid gas was given off, neither was there any hexachlorethane formed. He then added about 30 grms. of anhydrous chloride of aluminium, and again passed a current of dry chlorine as before. This time the gas was completely absorbed, and HCl was given off. After four hours the contents of the flask were solid. On throwing small portions of this white mass into water, hydrochloric acid is given off, and a white powder rises to the surface. This powder was dissolved in ether, the solution dried over chloride of calcium, and distilled. A body smelling strongly of camphor was obtained, sublimating at the ordinary temperature. Analysis showed that it was hexachlorethane (C_2Cl_6), which was formed according to the following equation: $-CCl_3, CCl_2H + Cl_2 = CCl_3, CCl_3 + HCl$. It is an excellent method of preparing hexachlorethane, and the return is almost theoretically exact.

Action of Chlorine on Tetrabromide of Acetylene in the presence of Chloride of Aluminium.—A. Mouneyrat.—Thirty grms. of anhydrous chloride of aluminium were added to 100 grms. of tetrabromide of acetylene, in a flask furnished with a vertical condenser and heated to 70–80°; hydrochloric acid is strongly given off. After four hours, a current of dry chlorine was passed, and at first more hydrochloric acid was given off, followed by torrents of bromine. When the latter ceased and no more chlorine was absorbed, the mass was thrown, a little at a time, into water containing soda-lye; a white powder came to the surface if the temperature had not exceeded 80°, and if the quantity of chloride of aluminium added was small; if, on the other hand, the temperature had exceeded 80°, and if the quantity of chloride of aluminium was in excess, the powder is black. After rectification, analysis showed this powder to be hexachlorethane, C_2Cl_6 , formed according to the following equation: $CHBr_2, CHBr_2 + Cl_2 = CCl_3, CCl_3 + Br_2 + (HCl)_2$.

On Glucosines.—C. Tanret.—The author finds that by acting on glucose by ammonia at 100° a series of bases is formed, among which are the glucosines α and β . MM. Brander and Stoehr contest the formulæ of these glucosines, maintaining that the glucosine α , boiling at 136°, has not the formula $C_6H_{12}N_2$, but is a mixture of pyridine, pyrazine, and methylpyrazine. As for the glucosine β , boiling at 155–160°, these authors only obtained a few decigrams of dimethylpyrazine boiling at 147–155°. M. Tanret shows that these conclusions are incorrect, and that the formulæ he gave are the true ones.

Hydrochlorate of Glucosamine.—C. Tanret.—The hydrochlorate of glucosamine possesses bi-rotation, but not always to the same extent. The author imagined that there must be modifications of this body, and he found that hydrochlorate of glucosamine- α had a rotatory power of $\alpha_D = +100^\circ$; and that the modification β , in a solution of 1 part in 2.5 parts of water gave $\alpha_D = 77.50^\circ$. The two modifications of hydrochlorate of glucosamine have a distinct rotatory power; and they, moreover, crystallise in two different systems.

On Active Methylbutylenediamine (Methyl-2, Diaminobutane 1.4),
 $NH_2CH_2CH(CH_3)CH_2NH_2$

—L. Etaix and P. Freundler.—The authors find that the successive operations to which they submitted the primitive body have not racemised it to any noticeable degree, though it might have been expected that either the action of hydrazin or of hydrochloric acid at 150° would destroy its rotatory power. It would appear from this that racemisation depends, not on the agents of transformation, but on the groupings which are attached to the asymmetric carbon.

On Dinitrophenyl-diacyetyl-methane.—F. Mottelet.—Acetylacetone of sodium is dissolved in strong alcohol in a water-bath, and to this solution, warm, is added an equal quantity of chloridinitrobenzene. A white crystalline deposit is immediately formed. When this deposit no longer increases the reaction is considered to have ended. The mass is thrown into water; an oil is deposited; this is acidulated slightly with hydrochloric acid, and the oil crystallises. The crystals are found by analysis to consist of dinitrophenyl-diacyetyl-methane.

Action of Chloride of Ethyloxalyl on Diphenyl in the presence of Chloride of Aluminium.—L. Rousset.—If small portions equal to one molecule of chloride of ethyloxalyl are dropped into a boiling sulphocarbonic solution containing one molecule of diphenyl and 133 grms. of chloride of aluminium, the theoretical quantity of hydrochloric acid is given off, and a brown mass, insoluble in sulphide of carbon, is left; this is treated with water, the sulphocarbonic layer is washed with water containing HCl, and then with water; the sulphide of carbon is driven off, and the residue purified in vacuo. It answers to the formula $C_6H_5, C_6H_4, CO, COOC_2H_5$.

Action of Chloride of Ethyl-oxalyl on Ethyl-naphthol in the presence of Chloride of Aluminium.—L. Rousset.—The condensation of chloride of ethyl-oxalyl with ethyl- α -naphthol is carried out in the same way as has previously been described for methyl- α -naphthol. The α -ethyloxynaphthylglyoxylic ether thus obtained boils at 240° to 245° under a pressure of 20 m.m. The glyoxylic acid which results from its saponification, by means of soda in aqueous solution, melts at 160°, and gives with aniline a phenylimide which crystallises in benzene in small yellow grains, fusible at 72°.

On the Perkin Reaction applied to some Aldehyde of the Naphthalene Series.—L. Rousset.—Not suitable for abstraction.

Products of Condensation of Saccharine with the Phenols.—P. Sisley.—Saccharine and resorcin heated together with sulphuric acid gives a yellowish brown

viscous mass. After cooling and throwing into water, yellow-brown crystals are formed; they are soluble in alcohol and acetic acid, but not in ether or chloroform, and they do not contain nitrogen. Its alkaline salts are very soluble, and possess a beautiful green dichroism; they dye silk in the same way as uranine. Analysis shows it to be identical with the sulphurine resorcin of Remsen. When treated with bromine in alcoholic solution it easily gives a bromised derivative forming red crystals, slightly soluble in water, and soluble in the alkalis, forming a red solution with a yellow fluorescence.

On Veratrylene-diamine.—Cl. Moureu.—Not suitable for abstraction.

Estimation of Phosphoric Acid.—H. Lasne.—Already inserted in full.

Apparatus for the Commercial Analysis of Gas.—L. Vignon.—A long paper, not suitable for abstraction.

Estimation of Small Quantities of Methyl-alcohol, Formic Aldehyd, and Formic Acid.—M. Nicloux.—Already inserted.

Cryoscopy of Milk.—A. Ponsot.—Hamburger found a variation of 0.013° for the congealing point of milk, with a mean value of -0.561, his results agreeing with those obtained by Winter; but when working with a Beckmann apparatus he found an average of -0.520° (-0.512° to -0.529°). A large part of this difference must be due to defective working,—for instance, he uses such a degree of cold that the milk, when introduced into the cryoscopic tube, is brought to a condition of surfusion. It is necessary that the same method should be followed by all, in order to get comparative results.

MEETINGS FOR THE WEEK.

TUESDAY, December 28th.

THURSDAY, December 30th.

SATURDAY, Jan. 1st, 1898

Royal Institution, 3. (Christmas Lectures). "Principles of the Electric Telegraph," by Prof. Oliver Lodge.

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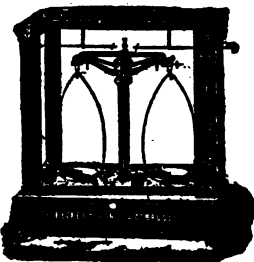
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Death.

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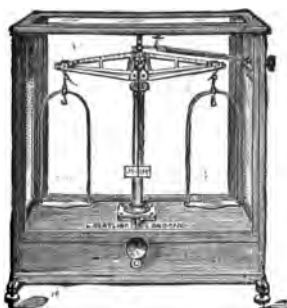
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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1988.

ON THE DENSITIES OF CARBONIC OXIDE, CARBONIC ANHYDRIDE, AND NITROUS OXIDE.*

By Lord RAYLEIGH, F.R.S.

THE observations here recorded were carried on by the method and with the apparatus described in a former paper ("On the Densities of the Principal Gases," *Roy. Soc. Proc.*, liii., p. 134, 1893), to which reference must be made for details. It must suffice to say that the globe containing the gas to be weighed was filled at 0° C., and to a pressure determined by a manometric gauge. This pressure, nearly atmospheric, is slightly variable with temperature on account of the expansion of the mercury and iron involved. The actually observed weights are corrected so as to correspond with a temperature of 15° C. of the gauge, as well as for the errors in the platinum and brass weights employed. In the present as well as in the former experiments I have been ably assisted by Mr. George Gordon.

Carbonic Oxide.

This gas was prepared by three methods. In the first method a flask, sealed to the rest of the apparatus, was charged with 80 grms. re-crystallised ferrocyanide of potassium and 360 c.c. strong sulphuric acid. The generation of gas could be started by the application of heat, and with care it could be checked and finally stopped by the removal of the flame with subsequent application, if necessary, of wet cotton-wool to the exterior of the flask. In this way one charge could be utilised with great advantage for several fillings. On leaving the flask the gas was passed through a bubbler containing potash solution (convenient as allowing the rate of production to be more easily estimated), and thence through tubes charged with fragments of potash and phosphoric anhydride, all connected by sealing. When possible, the weight of the globe full was compared with the mean of the preceding and following weights empty. Four experiments were made with results agreeing to within a few tenths of a m.grm.

In the second set of experiments the flask was charged with 100 grms. of oxalic acid and 500 c.c. strong sulphuric acid. To absorb the large quantity of CO₂ simultaneously evolved a plentiful supply of alkali was required. A wash-bottle and a long nearly horizontal tube contained strong alkaline solution, and these were followed by the tubes containing solid potash and phosphoric anhydride as before.

For the experiments of the third set oxalic acid was replaced by formic, which is more convenient as not entailing the absorption of large volumes of CO₂. In this case the charge consisted of 50 grms. formate of soda, 300 c.c. strong sulphuric acid, and 150 c.c. distilled water. The water is necessary in order to prevent action in the cold, and the amount requires to be somewhat carefully adjusted. As purifiers, the long horizontal bubbler was retained, and the tubes charged with solid potash and phosphoric anhydride. In this set there were four concordant experiments. The immediate results stand thus:—

Carbonic Oxide.

From ferrocyanide	2.29843
" oxalic acid	2.29852
" formate of soda	2.29854
Mean	2.29850

* A Paper read before the Royal Society, December 9th, 1897.

This corresponds to the number 2.62704 for oxygen (*loc. cit.*, p. 144), and is subject to a correction (additive) of 0.00056 for the diminution of the external volume of the globe when exhausted.

The ratio of the densities of carbonic oxide and oxygen is thus 2.29906 : 2.62760; so that if the density of oxygen be taken as 32, that of carbonic oxide will be 27.9989. If, as some preliminary experiments by Dr. Scott (*Camb. Phil. Proc.*, ix., p. 144, 1896) indicate, equal volumes may be taken as accurately representative of CO and of O₂, the atomic weight of carbon will be 11.9989 on the scale of oxygen = 16.

The very close agreement between the weights of carbonic oxide prepared in three different ways is some guarantee against the presence of an impurity of widely differing density. On the other hand, some careful experiments led Mr. T. W. Richards (*Amer. Acad. Proc.*, xviii., p. 279, 1891) to the conclusion that carbonic oxide is liable to contain considerable quantities of hydrogen or of hydrocarbons. From 5½ litres of carbonic oxide passed over hot cupric oxide he collected no less than 25 m.grms. of water, and the evidence appeared to prove that the hydrogen was really derived from the carbonic oxide. Such a proportion of hydrogen would entail a deficiency in the weight of the globe of about 11 m.grms., and seems improbable in view of the good agreement of the numbers recorded. The presence of so much hydrogen in carbonic oxide is also difficult to reconcile with the well-known experiments of Professor Dixon, who found that prolonged treatment with phosphoric anhydride was required in order to render the mixture of carbonic oxide and oxygen inexplorable. In the presence of relatively large quantities of free hydrogen (or hydrocarbons) why should traces of water vapour be so important?

In an experiment by Dr. Scott (*Chem. Soc. Trans.*, 1897, p. 564) 4 litres of carbon monoxide gave only 1.3 m.grms. to the drying tube after oxidation.

I have myself made several trials of the same sort with gas prepared from formate of soda exactly as for weighing. The results were not so concordant as I had hoped,* but the amount of water collected was even less than that given by Dr. Scott. Indeed I do not regard as proved the presence of hydrogen at all in the gas that I have employed.†

Carbonic Anhydride.

This gas was prepared from hydrochloric acid and marble, and, after passing a bubbler charged with a solution of carbonate of soda, was dried by phosphoric anhydride. Previous to use, the acid was caused to boil for some time by the passage of hydrochloric acid vapour from a flask containing another charge of the acid. In a second set of experiments the marble was replaced by a solution of carbonate of soda. There is no appreciable difference between the results obtained in the two ways; and the mean, corrected for the errors of weights and for the shrinkage of the globe when exhausted, is 3.6349, corresponding to 2.6276 for oxygen. The temperature at which the globe was charged was 0° C., and the actual pressure that of the manometric gauge at about 20°, reduction being made to 15° by the use of Boyle's law. From the former paper it appears that the actual height of the mercury column at 15° is 762.511 m.m.

Nitrous Oxide.

In preliminary experiments the gas was prepared in the laboratory, at as low a temperature as possible, from nitrate of ammonia, or was drawn from the iron bottles in which it is commercially supplied. The purification was by passage over potash and phosphoric anhydride. Unless

* One obstacle was the difficulty of re-oxidising the copper reduced by carbonic oxide. I have never encountered this difficulty after reduction by hydrogen.

† In Mr. Richards's work the gas, in an imperfectly dried condition, was treated with hot platinum-black. Is it possible that the hydrogen was introduced at this stage?

special precautions are taken the gas so obtained is 10 or more m.grms. too light, presumably from admixture with nitrogen. In the case of the commercial supply a better result is obtained by placing the bottles in an inverted position so as to draw from the liquid rather than from the gaseous portion.

Higher and more consistent results were arrived at from gas which had been specially treated. In consequence of the high relative solubility of nitrous oxide in water, the gas held in solution after prolonged agitation of the liquid with impure gas from any supply will contain a much diminished proportion of nitrogen. To carry out this method on the scale required, a large (11-litre) flask was mounted on an apparatus in connection with the lathe so that it could be vigorously shaken. After the dissolved air had been sufficiently expelled by preliminary passage of N_2O , the water was cooled to near $0^\circ C.$, and violently shaken for a considerable time while the gas was passing in large excess. The nitrous oxide thus purified was expelled from solution by heat, and was used to fill the globe in the usual manner.

For comparison with the results so obtained, gas purified in another manner was also examined. A small iron bottle, fully charged with the commercial material, was cooled in salt and ice, and allowed somewhat suddenly to blow off half its contents. The residue drawn from the bottle in one or other position was employed for the weighings.

Nitrous Oxide (1896).

Aug. 15	Expelled from water	3.6359
" 17	"	3.6354
" 19	From residue after blow off,	
	valve downwards	3.6364
" 21	Ditto " upwards	3.6358
" 22	Ditto " downwards	3.6360
Mean		3.6359

The mean value may be taken to represent the corrected weight of the gas which fills the globe at $0^\circ C.$ and at the pressure of the gauge (at 15°), corresponding to 2.6276 for oxygen.

One of the objects which I had in view in determining the density of nitrous oxide was to obtain, if it were possible, evidence as to the atomic weight of nitrogen. It may be remembered that observations upon the density of pure nitrogen, as distinguished from the atmospheric mixture containing argon which, until recently, had been confounded with pure nitrogen (Rayleigh and Ramsay, *Phil. Trans.*, vol. clxxxvi., p. 190, 1895), led to the conclusion that the densities of oxygen and nitrogen were as 16 : 14.003, thus suggesting that the atomic weight of nitrogen might really be 14 in place of 14.05, as generally received. The chemical evidence upon which the latter number rests is very indirect, and it appears that a direct comparison of the weight of nitrous oxide and of its contained nitrogen might be of value. A suitable vessel would be filled, under known conditions, with the nitrous oxide, which would then be submitted to the action of a spiral of copper or iron wire rendered incandescent by an electric current. When all the oxygen was removed, the residual nitrogen would be measured, from which the ratio of equivalents could readily be deduced. The fact that the residual nitrogen would possess nearly the same volume as the nitrous oxide from which it was derived would present certain experimental advantages. If indeed the atomic weights were really as 14 : 16, the ratio (x) of volumes, after and before operations, would be given by—

$$\frac{2.2996 \times x}{3.6359 - 2.2996 \times x} = \frac{14}{8},$$

whence—

$$x = \frac{7 \times 3.6359}{11 \times 2.2996} = 1.0061,$$

3.6359 and 2.2996 being the relative weights of nitrous oxide and of nitrogen which (at $0^\circ C.$ and at the pressure

of the gauge) occupy the same volume. The integral numbers for the atomic weights would thus correspond to an expansion, after chemical reduction, of about one-half per cent.

But in practical operation the method lost most of its apparent simplicity. It was found that copper became unmanageable at a temperature sufficiently high for the purpose, and recourse was had to iron. Coils of iron suitably prepared and supported could be adequately heated by the current from a dynamo without twisting hopelessly out of shape; but the use of iron leads to fresh difficulties. The emission of carbonic oxide from the iron heated in vacuum continues for a very long time, and the attempt to get rid of this gas by preliminary treatment had to be abandoned. By final addition of a small quantity of oxygen (obtained by heating some permanganate of potash sealed up in one of the leading tubes) the CO could be oxidised to CO_2 , and thus, along with any H_2O , be absorbed by a lump of potash placed beforehand in the working vessel. To get rid of superfluous oxygen, a coil of incandescent copper had then to be invoked, and thus the apparatus became rather complicated.

It is believed that the difficulties thus far mentioned were overcome, but nevertheless a satisfactory concordance in the final numbers was not attained. In the present position of the question no results are of value which do not discriminate with certainty between 14.05 and 14.00. The obstacle appeared to lie in a tendency of the nitrogen to pass to higher degrees of oxidation. On more than one occasion mercury (which formed the movable boundary of an overflow chamber) was observed to be attacked. Under these circumstances I do not think it worth while to enter into further detail regarding the experiments in question.

The following summary gives the densities of the various gases relatively to air, all obtained by the same apparatus (*Roy. Soc. Proc.*, vol. liii., p. 148, 1893; vol. lv., p. 340, 1894; *Phil. Trans.*, vol. clxxxvi., p. 189, 1895; *Roy. Soc. Proc.*, vol. lix., p. 201, 1896). The last figure is of little significance:—

Air free from H_2O and CO_2	1.00000
Oxygen	1.10535
Nitrogen and argon (atmospheric) ..	0.97209
Nitrogen	0.96737
Argon	1.37752
Carbonic oxide	0.96716
Carbonic anhydride	1.52909
Nitrous oxide	1.52951

The value obtained for hydrogen upon the same scale was 0.06960; but the researches of M. Leduc and of Professor Morley appear to show that this number is a little too high.

ELECTRIC CONDUCTIVITY OF NITRIC ACID.*

By V. H. VELEY, M.A., F.R.S., and
J. J. MANLEY, Dabney Curator of the Magdalen College
Laboratory, Oxford.

In this paper an account is given of determinations of the electric conductivity of nitric acid of percentage concentrations varying from 1.3 to 99.97, purified, so far as possible, from reduction products of the acid, as also from sulphuric and the halogen acids, with which it is likely to be contaminated from its process of manufacture. In the preliminary experiments it was observed that the results might be vitiated by (1) a trace of nitrous acid either directly acid or produced by decomposition due to exposure to sunlight, and (2) imperfect insulation of the

* Abstract of a Paper read before the Royal Society, December 9th, 1897.

electrolytic cell caused by metallic clamps, a point which seems to have been neglected by previous observers.

The methods adopted for the purification of the water and nitric acid, as also for the detection and estimation of the impurities, are described in full. The greatest quantity of nitrous acid, sulphuric acid, and the halogen acids found in any sample used were 0.75, 4.3, and 3.8 parts per million respectively.

The thermometers, resistance coils, and other instruments used, were compared with certain standards and corrected accordingly; the burettes and electrolytic cells were calibrated by one or more methods, and the mean of the values accepted.

The method adopted for the determinations was in outline that originally described by Kohlrausch, but modified so as to overcome certain difficulties experienced. A particular form of bridge was constructed, in which the wire was an air line, and a special form of slider adopted to tap without sagging the wire, so arranged that it could be moved by the observer from the extremity of the bridge, and thus all thermo-currents due to his proximity were avoided.

A rapidly revolving commutator was substituted for the usual induction coil, as the latter was found to be unsatisfactory owing to the susceptibility of nitric acid to polarisation.

Various forms of electrolytic cells were used according to the concentration of the acid and the temperature of the observations; these were provided with movable electrodes, so as to throw into circuit different lengths of acid.

A special form of apparatus was devised to prepare nitric acid of 99.88 per cent, and another form to obtain acid of 99.97 per cent from the latter. As a considerable quantity of this practically anhydrous acid was obtained, its chemical and certain physical properties were examined. It has no action on (1) copper, (2) silver, (3) cadmium, and (4) mercury, all of high degree of purity, and (5) commercial magnesium, at ordinary temperatures; purified iron and commercial granulated tin were unaffected by the acid, even when boiling. Purified zinc was slightly acted upon, but sodium immediately caught fire. The acid has no action whatever on calcium carbonate at ordinary temperatures or the boiling-point. Flowers of sulphur and iron pyrites dissolve quickly and completely in the gently warmed acid. The following results were obtained for the density of the 99.97 per cent acid, corrected for weighings *in vacuo* :—

Density $4/4 = 1.54212$; $14/24 = 1.52234$; $24/24 = 1.50394$, the mean values of two concordant observations.

As a further check upon the measurements obtained by the Kohlrausch method, certain other measurements were made by Carey Foster's method for the comparison of resistances, and the results obtained were found to be concordant within the limits of experimental error. In a series of tables the values are given for thirty-two samples of acid of the specific resistance in true ohms at temperatures of 0°, 15°, and 30°, the temperature coefficients α_{10° and β_{10° deduced from the equation—

$$R_t = R_0(1 + \alpha t - \beta t^2),$$

as also for $K_0 \times 10^4$, $K_{15} \times 10^4$, and $K_{30} \times 10^4$ (the conductivity of mercury at 0° being taken as unity, and its specific resistance as 94.07 microhms per 1 c.c.).

It is shown that the specific resistance decreases for percentage concentrations from 1.30 to 30, at first more, then less rapidly (thus confirming the previous observations of Kohlrausch); from this point the resistance increases slowly up to 76 per cent, thence more rapidly until a maximum is reached at 96.12 per cent, when a sudden reversal takes place.

Further, whereas nitric acid behaves as other electrolytes in possessing a positive temperature coefficient of conductivity for percentage concentrations from 1.3 to 96.12, yet from this point up to 99.97 per cent it behaves

as a metallic conductor in possessing a negative temperature coefficient.

Similar phenomena have been observed by Arrhenius in the cases of moderately dilute solutions of hypophosphorous and phosphoric acids, and explained by him by means of the ionic dissociation hypothesis. It is pointed out that nitric acid of 96 to 99.97 per cent would, *ex hypothesi*, contain few, if any, free ions, and therefore the theory would lead to a totally opposite conclusion.

The results of the experiments are also discussed in relation to the hydrate theory of solution, and the illustrative curves in which the percentages of acid are taken as abscissae, and the resistances or conductivities in mercury units, show points of discontinuity markedly at percentages corresponding approximately to the composition required for the hydrates $\text{HNO}_3 \cdot 2\text{H}_2\text{O}$, $\text{HNO}_3 \cdot \text{H}_2\text{O}$, $2\text{HNO}_3 \cdot \text{H}_2\text{O}$ ($= \text{H}_4\text{N}_2\text{O}_7$), and less markedly for the hydrate $\text{HNO}_3 \cdot 10\text{H}_2\text{O}$. Further, if the values of $\alpha \times 10^4$ and $\beta \times 10^4$ are referred to molecular proportions of water, the minima values of the former and the maxima of the latter occur in the cases of 3.07, 1.84, 0.99, and 0.55 molecular proportions or very approximately—

$\text{HNO}_3 \cdot 3\text{H}_2\text{O}$, $\text{HNO}_3 \cdot 2\text{H}_2\text{O}$, $\text{HNO}_3 \cdot \text{H}_2\text{O}$, and $2\text{HNO}_3 \cdot \text{H}_2\text{O}$.

Further evidence is thus added by an independent method to that already accumulated as to the existence of definite combination of nitric acid with water. Finally, it is pointed out that if a curve is plotted out in which the molecular proportions of water are taken as abscissae, and the values for $\alpha \times 10^4$ as ordinates, there are ascending and descending branches, meeting at the points corresponding to the formation of the respective hydrates; the phenomena are compared with those observed by Bakhuis-Roozeboom for the solubility curves of hydrates of ferric chloride and by Le Chatelier, as also by Heycock and Neville for the freezing-point of alloys.

ON THE OCCLUSION OF HYDROGEN AND OXYGEN BY PALLADIUM.*

By LUDWIG MOND, Ph.D., F.R.S.,
WILLIAM RAMSAY, Ph.D., LL.D., Sc.D., F.R.S., and
JOHN SHIELDS, D.Sc., Ph.D.

DURING their investigations on the nature of the occlusion of gases by finely divided metals, and in particular on the occlusion of hydrogen and oxygen by platinum black, the authors have had occasion to examine the behaviour of palladium to these gases.

The palladium was employed in three states of aggregation, viz., in the form of (a) black, (b) sponge, and (c) foil. Palladium black, prepared in the same way as platinum black, contains 1.65 per cent of oxygen, or, taking the density of palladium black as 12.0, 138 volumes of oxygen. It differs from platinum black, however, inasmuch as the oxygen cannot be removed *in vacuo* at a dull red heat, and consequently had to be determined in the ignited substance by passing hydrogen over it and weighing the water produced. Palladium black dried at 100° contains 0.72 per cent of water, and hence, on the assumption that the oxygen exists as PdO, we have for the analysis of palladium black :—

Pd	86.59 per cent.	
PdO	12.69 ..	= 1.65 per cent O ₂ .
H ₂ O	0.72 ..	

On heating in an atmosphere of oxygen, palladium black goes on absorbing oxygen at least up to a red heat, with the formation of a brownish black substance, which does not again lose its oxygen at a dull red heat *in vacuo*.

* Abstract of a Paper read before the Royal Society, December 16th, 1897.

The amount of oxygen absorbed (nearly 1000 volumes) was about one and a half times as much as corresponds with the formula Pd_2O ; and if the ignition had been sufficiently prolonged, the whole of the palladium would probably have been converted into the oxide PdO .

Palladium black, when exposed to hydrogen gas, absorbed over 1100 volumes, but of this only 873 volumes were really occluded, the remainder having formed water with 139 volumes of oxygen originally contained in the black, which is in good agreement with the direct gravimetric estimation.

Of the hydrogen occluded, about 92 per cent was pumped off slowly at the ordinary temperature, and almost the whole of the remainder at 444° . Increase of pressure of the hydrogen from one atmosphere up to 4.6 atmospheres had no influence on the quantity occluded at the ordinary temperature.

The pure palladium sponge remaining in the experimental tube after the above experiment was over occluded 852 volumes of hydrogen, and about 98 per cent of this was extracted *in vacuo* at the ordinary temperature.

New palladium foil behaved in a very peculiar fashion. At first it scarcely occluded any hydrogen even after ignition in the gas and subsequently cooling down. It was therefore charged and discharged several times electrolytically with hydrogen, but still it persistently refused to occlude any appreciable quantity when replaced in an atmosphere of hydrogen.

After powerful ignition in the blowpipe flame, when it was probably oxidised and then again reduced at a still higher temperature, it was introduced once more into the experimental tube. It immediately occluded a considerable quantity of hydrogen, and by maintaining the temperature between 100° and 130° a large additional quantity was slowly absorbed. On cooling down to the ordinary temperature, hydrogen was again occluded, and it was finally found to have taken up 846 volumes, *i.e.*, approximately the same quantity as the black or sponge.

The hydrogen occluded by palladium foil is given off again very slowly at the ordinary temperature *in vacuo*, but rapidly and almost completely at 100° .

The paper contains some attempts to explain the extraordinary behaviour of palladium foil.

The heat evolved on the occlusion of hydrogen by palladium black was measured in an ice calorimeter (temperature of the room $20-24^\circ$) in nearly the same way as the corresponding heat of occlusion of hydrogen by platinum black, thereby avoiding errors due to the pre-existence of oxygen in the substance.

Favre's statement that the heat of occlusion remains constant for the different fractions of hydrogen occluded was confirmed, and it was found that $+46.4$ K (4640 g. cal.) were evolved per grm. of hydrogen occluded.

The authors consider that this number may be taken as correct within 1 per cent, and compare it with the different values found by Favre and those calculated by Moutier and Dewar.

If the external work done by the atmosphere be eliminated, the heat evolved per grm. of hydrogen occluded becomes $+43.7$ K.

The heat evolved per grm. of oxygen absorbed was also determined in an indirect manner, and found to be $+11.2$ K (1120 g. cal.).

This number, referred to 16 grms. of oxygen, lies intermediate between the values given by Thomsen for the heat of formation of palladious and palladic hydroxides, and may be consistent, considering the accuracy of such measurements, with the formation of either of these hydroxides or with a mixture of both. In any case it is of the same order of magnitude, and taken in conjunction with the behaviour of palladium black when heated in an atmosphere of oxygen, is undoubtedly in harmony with the view that the absorption of oxygen by palladium black (and probably also by platinum black) is a true phenomenon of oxidation.

The authors have also investigated the atomic ratio—

palladium : hydrogen for fully-charged palladium black, sponge, and foil, and give in tabular form the corresponding ratios deduced from experiments by Graham and Dewar in which wire and block palladium were charged with hydrogen electrolytically. They have arrived at the conclusion that no matter whether the palladium exists as black, sponge, foil, wire, or compact metal, or whether it is charged by direct exposure to hydrogen gas (the proper conditions being observed), or charged electrolytically, the amount of hydrogen occluded in each case is approximately the same, the atomic ratio varying between 1.37 and 1.47.

Hoitsema has shown that Troost and Hautesfeuille's deduction that a compound exists having the formula Pd_2H is not warranted. The constancy of the heat of occlusion over the whole range of absorption is also opposed to the view that such a compound is formed.

The composition of fully charged palladium hydrogen corresponds closely with the formula Pd_2H_2 first suggested by Dewar. The principal and almost only evidence, up to the present, in favour of the formation of such a definite chemical compound is to be found in the approximation of the above atomic ratios to the theoretical value 1.5, required by the formula Pd_2H_2 . Although Hoitsema's arguments may be equally well directed against the existence of this compound, the authors consider that additional and independent evidence is desirable, and hope to be able to provide it.

It is also shown that the heats of occlusion of hydrogen in platinum and palladium black are not in favour of the view which has sometimes been put forward that the heat of occlusion of a gas represents the heat of condensation or liquefaction of the gas in the capillary pores of the absorbing substance or the heat of solidification or fusion.

"ON FEHLING'S SOLUTION."

By OTTO ROSENHEIM, Ph.D., F.C.S., and
PHILIP SCHIDROWITZ, Ph.D., F.C.S.

THE *Berichte der Deutschen Chem. Gesellschaft* (vol. xxx., p. 2431) contains a paper by M. Z. Jovitschitch under the above heading, in which the author asserts that, according to an observation made by Professor Siegfried, mineral acids (hydrochloric, nitric, and sulphuric) possess the property of reducing Fehling's solution—more especially when the latter is still alkaline. Granted that the author's observations are correct, it follows that the reduction is due to the alkali salts of the acids mentioned. As salts of the alkalis are almost invariably present in liquids in which matter capable of oxidation is to be tested for or determined, the experimental confirmation of Jovitschitch's statements seemed to us to be highly desirable, especially as no analytical data were recorded in his paper.

Viewed theoretically, there is absolutely no reason why salts of the alkalis should influence the reduction of Fehling's solution; and, as might have been expected, both qualitative and quantitative experiments made by us yielded negative results.* It must be assumed that Jovitschitch's paper is based on an error of observation, and as T. E. Gerock (*Ber.*, xxx., 2865) has meanwhile come to the same conclusion, we refrain from discussing the subject more fully, and merely record the data of some quantitative experiments which are directly to the point. The method adopted was similar to that usually employed in the gravimetric estimation of sugars. Fifty

* Considering that Fehling's solution (in various modifications) has now been in use for close on fifty years, it is indeed remarkable that the phenomena described in the paper quoted have not been observed before. On the contrary, Boivin and Loiseau (*Ber.*, vii., 1790) assert that the presence of small quantities of salts, such as CaCl_2 , BaCl_2 , NH_4Cl , NaCl , &c., prevents the spontaneous reduction that takes place on boiling the solution with distilled water.

c.c. of Fehling's solution (stored in two solutions and mixed when required), diluted with 25 c.c. of distilled water, were heated to boiling, 25 c.c. of a 25 per cent solution of the salt in question run in from a pipette, and after ebullition had re-commenced, the liquid was kept boiling in one series for two, in the other for four minutes. The perfectly clear solution was filtered hot through a Soxhlet's asbestos tube under reduced pressure, the tube dried, heated in hydrogen in the usual manner, and finally weighed. As will be seen from the figures below, the results were practically identical with those obtained by the boiling of Fehling's solution alone.

	Boiled for two minutes. Grm. Cu.	Boiled for four minutes. Grm. Cu.
Fehling's solution alone* ..	0'0028	0'0040
25 per cent potassium chloride ..	0'0023	0'0040
25 per cent potassium nitrate ..	0'0036	0'0044
Potassium sulphate (saturated at 18° C.)	0'0020	0'0040

ON THE NON-EXISTENCE OF AN INTERMEDIATE IODIDE OF MERCURY.

By MAURICE FRANCOIS.

BEFORE 1827 only two iodides of mercury were accepted—the yellowish green mercurous iodide and the red mercuric iodide. At this date Boullay described the intermediate iodide, which he simply called the yellowish iodide, calling the protoxide simply green (*Ann. de Chim. et de Phys.* [2], xxxiv., p. 364, 1827). This new compound of the atomic formula, Hg_4I_6 , has been described as sublimable without decomposition, and as insoluble in boiling alcohol. Boullay has given for its preparation a method, since quoted in all standard chemical works. It consists in adding to a slightly acid solution of mercurous nitrate a solution of iodide of potassium, to which has been added a quantity of free iodine, equal to half the amount it already contains in the state of iodide. Such an operation would undoubtedly give a product of the desired composition, on condition that the precipitation was complete, and that its composition was not changed by the washings. But Boullay recommended stopping the addition of iodide of potassium at the moment when the precipitate, originally yellow, takes a red tint.

I have endeavoured to verify the facts put forward by Boullay, facts which have not up till the present time been contested, and I have arrived at results completely opposed to those of this *savant*. For this research I prepared the compound described by Boullay, by not stopping the addition of the iodised iodide until the precipitate was ended. The precipitate, well washed with water, was collected, analysed, and submitted to further washings with pure ether. I also fractionated the precipitate and analysed the different fractions.

1. I prepared a solution of iodised potassic iodide, containing 166 grms. of iodide of potassium and 63'50 grms. of free iodine per litre. I also made, with freshly prepared and well-dried mercurous nitrate, a solution containing 40 grms. of mercurous nitrate, and 2 c.c. of pure nitric acid per 400 c.c. The iodised solution was put into a burette and allowed to fall slowly, drop by drop, into the solution of mercurous nitrate, which was kept constantly stirred with a glass rod. Each drop made a red splash, which rapidly became yellow.

The colour of the precipitate first formed is of a beautiful yellow; then, as we continue to add the iodised

solution, it passes to a reddish orange, and finally to red. We stop the addition when a drop of the iodised iodide gives no further precipitate in the clear solution, but only a yellow colouration. At this moment we have used 132 c.c. of the iodised solution.

The whole of the precipitate is now red; it is washed with water by decantation, and dried at 60°. It weighs 51 grms. Its composition is as follows:—

Mercury	50'63 per cent.
Iodine	48'50 "

The theoretical composition of the intermediate iodide is:—

Mercury	51'21 per cent.
Iodine	48'79 "

As I stated above, the composition of this precipitate corresponds closely with that of the intermediate iodide.

This precipitate was then washed with cold ether by decantation, in the dark. As I have said elsewhere, this is a method of separating mercurous from mercuric iodide, which is much more exact than that of washing with boiling alcohol, being almost irreproachable. To exhaust 1 gm. of intermediate iodide we use three washings of 50 c.c. each of pure ether, decant each time on a filter, and evaporate the ether under a bell-jar over sulphuric acid, in a small tared crystallising glass. This treatment has removed, from 1 gm. of intermediate iodide, 0'591 gm. of mercuric iodide. The residue, insoluble in ether, is of a very pale yellow colour, and weighs 0'411 gm. Its composition is as follows:—

Mercury	60'85 per cent.
Iodine	38'70 "

The theoretical composition of mercurous iodide is:—

Mercury	61'16 per cent.
Iodine	38'84 "

Thus the so-called intermediate iodide can be split up by ether into mercurous and mercuric iodides.

2. To see better what variations the precipitate undergoes during the gradual addition of the iodised iodide, I carried out a fractional precipitation in the following manner:—

I operated on 40 grms. of mercurous nitrate dissolved in 400 c.c. of water and 20 c.c. of nitric acid. Knowing already that this quantity requires for complete precipitation 132 c.c. of the iodised solution, I used 13'2 c.c. of the latter for each precipitation, so as to obtain ten fractions of the precipitate. The affusion of the iodised iodide is done drop by drop while agitating rapidly.

The first precipitate is thrown on a filter, air-dried with a filter-pump, and the filtrate treated as before for the second precipitate, and so on to the end. Each precipitate is carefully washed with water, and dried at 60°.

Analysis gave the following figures:—

- 1st fraction.—Pure bright yellow colour. Mercury 61'02 per cent. Iodine 38'66 per cent.
- 2nd fraction.—Pure bright yellow colour. Mercury 61'29 per cent. Iodine 38'50 per cent.
- 3rd fraction.—Bright yellow. Mercury 61'19 per cent. Iodine 38'19 per cent.
- 4th fraction.—Bright red. Mercury 44'23 per cent. Iodine 55'32 per cent.
- 5th fraction.—Bright red. Mercury 44'81 per cent. Iodine 54'55 per cent.
- 6th fraction.—Bright red. Mercury 44'95 per cent. Iodine 54'80 per cent.
- 7th fraction.—Bright red. Mercury 45'72 per cent. Iodine 53'65 per cent.
- 8th fraction.—Bright red. Mercury 45'93 per cent. Iodine 54'00 per cent.
- 9th fraction.—Bright red. Mercury 44'51 per cent. Iodine 55'02 per cent.
- 10th fraction.—Bright red. Mercury 44'30 per cent. Iodine 55'56 per cent.

* It is a well-known fact that even the most carefully prepared Fehling's solution gives a slight precipitation of cuprous oxide on heating, varying in amount from 2 to 3 m.grms. (see, also, Brown, Morris, and Millar, *Journ. Chem. Soc.*, lxxi., 96).

The theoretical composition of mercurous iodide is:—

Mercury	61.16 per cent.
Iodine	38.84 „

and that of mercuric iodide:—

Mercury	44.05 per cent.
Iodine	55.55 „

As is easily seen, the first three fractionations are of pure mercurous iodide, the others of fairly pure mercuric iodide. It is worthy of remark that the mercurous iodide obtained in this manner is of a very pure yellow colour.

This reaction, and the successive appearance of the mercurous and mercuric iodides, can be explained by the two following experiments.

When we put about 1 grm. of mercuric iodide, recently precipitated and washed but not dried,—that is to say, in the finest possible state of division,—into 100 c.c. of our mercurous nitrate solution at 40 grms. per 400 c.c., we notice, after about fifteen minutes, that the red mercuric iodide changes to a pure yellow colour, being transformed into mercurous iodide. There is an increase in weight of the iodide, and the reaction may be expressed thus:—



This is the transformation of the red splash into yellow, already mentioned above.

When, on the contrary, we put 1 grm. of mercurous iodide into 100 c.c. of a solution of mercuric nitrate containing 40 grms. of nitrate per 400 c.c., we notice that, after about fifteen minutes, the yellow mercurous iodide changes into the red mercuric iodide, according to the equation—



There are thus two limiting contrary reactions.

But when we pour the iodised solution of potassium iodide into mercuric nitrate, the free iodine produces mercuric nitrate, since the mercury of the mercurous nitrate cannot be precipitated except by producing mercuric nitrate or nascent oxygen, which comes to the same thing.

We have thus a liquid in which the proportion of mercurous nitrate goes on diminishing, while the proportion of mercuric nitrate goes on increasing: this sufficiently explains why the nature of the precipitate changes at a given moment, and why the precipitate is at first mercurous iodide and afterwards mercuric iodide. This oxidising action of free iodine seems to have escaped Boullay.

It seems extraordinary that a chemist of such repute as Boullay should have been mistaken on this point. Being fully persuaded that mercurous iodide is green, and the greener it is the purer it is, he could not admit that a distinctly yellow substance was mercurous iodide. Further, he was at this time much preoccupied in proving the existence of double iodides, in which one of the iodides plays the part of an acid, and the other the part of a base; the existence of an intermediate iodide fitted in with his theory, for he regarded it as a combination of mercurous and mercuric iodides. One feels convinced after reading Boullay's paper that he established the existence of this body by relying on his theoretical notions, and on the colour more than on analysis, for he only gives one estimation of mercury, by means of metallic iron, the iodine being estimated by difference. Moreover, the substance analysed by him had not been previously purified.

Conclusions.—In attempting to prepare the intermediate iodide by Boullay's method (1), we obtain a mixture of mercurous and mercuric iodides, separated by ether; (2), the first portions of the precipitate are pure mercurous iodide, to which succeeds pure mercuric iodide.

The intermediate iodide of mercury is therefore not a chemical compound, but a mixture, the more yellow and

rich in mercurous iodide in proportion as the precipitation has been pushed less far.—*Journ. de Pharm. et de Chim.*, Series 6, vol. vi., No. 10, 1897.

ON CERIUM.

By O. BOUDOUARD.

I HAVE the honour of submitting to the Academy the results of researches on the salts of cerium. Continuing the work which I have undertaken with my regretted master, Paul Schützenberger, I have chiefly studied the cerium acetate and sulphate.

Cerium Acetate.

174 grms. cerium sulphate, *free from thorium*, are dissolved in water and treated with the corresponding quantity of lead acetate, to obtain cerium acetate. The excess of lead is removed by means of a current of hydrogen sulphide. After filtering off the lead sulphide, the ceric solution is allowed to settle in the cold, and in a very short time deposits an abundant white precipitate.

A portion of this acetate was converted into sulphate, and the sulphate was analysed by ignition. I have thus found the atomic weight of the corresponding metal—

$$\text{Ce} = 137.85.$$

Having made fractionated crystallisations of this sulphate, I obtained—

First crystallisation	Ce = 140.7
Mother-liquor	Ce = 138.5

In all the crystallisations which have been effected the mother-liquors were always precipitated by alcohol. The sulphate thus obtained, after desiccation, is dissolved in cold water and again crystallised; this precaution is taken in order to obtain salts absolutely neutral.

The clear solution obtained from the filtration of the basic acetate was concentrated in the water-bath. The first deposit formed gave on analysis:—

First deposit .. {	Crystallisation (a) ..	Ce = 137.35
	Mother-liquors (a) ..	Ce = 135.1

Proceeding then with a series of fractionated crystallisations, I obtained the following results:—

Second deposit {	Crystals (b)	Ce = 136.5
	Mother-liquors (b) ..	Ce = 137.4
Mother-liquors. {	Crystals (c)	Ce = 139.1
	Mother-liquors (c) ..	Ce = 136.05

Using Peroxide of Hydrogen.—If to a solution of acetate of cerium we add an excess of peroxide of hydrogen, a yellow precipitate is formed; this precipitation is facilitated by heat, which, however, should not be kept up for too long a time; and, further, the precipitation is only partial.

In an experiment I made, I obtained 6 grms. of oxide, which was transformed into sulphate and submitted to fractional crystallisations:—

First crystallisation	Ce = 137.15
Mother-liquor	Ce = 137.6

The oxides were mixed afresh, transformed into sulphates, and these submitted to another fractional crystallisation, which gave—

First crystallisation	Ce = 137.15
Second	Ce = 137.35
Third	Ce = 137.6

Oxalic acid was added to the part not precipitated by peroxide of hydrogen. The oxalate of cerium was calcined and the oxide transformed into sulphate. Fractional crystallisations gave—

First crystallisation	Ce = 137.85
Second	Ce = 139.9
Third	Ce = 138.85

Sulphate of Cerium.

A solution of cerous sulphate, to which had been added 20 grms. of sulphate of potassium, produced a double sulphate, which I will call S.D. No. 1. The clear liquid was re-precipitated by means of an equal quantity of sulphate of potassium; this gave me the precipitate S.D. No. 2. Continuing in this manner, I obtained S.D. No. 3 and S.D. No. 4; the wash waters from this last precipitate contained nothing further. Each of these double sulphates was decomposed by caustic soda; the hydrate obtained was washed with warm water, and finally dissolved in nitric acid and precipitated by oxalic acid. This oxalate was calcined, and the oxide transformed into sulphate. I thus made a series of crystallisations with the following results:—

S.D. No. 1.—First crystallisation..	Ce=138.75
Second	Ce=137.3
Third	Ce=133.0
S.D. No. 2.—First	Ce=138.5
Second	Ce=136.95
Third	Ce=137.9
Fourth	Ce=137.7
S.D. Nos. 3 and 4.—First crystallisation	Ce=138.25
Second	Ce=136.25

All these results, whether obtained with acetate or sulphate of cerium, show that, conformably with the indications already given by Schützenberger (*Comptes Rendus*, cxx., p. 962), oxide of cerium is accompanied by small quantities of another earth with a lower atomic weight. This earth should be susceptible of giving a binoxide by oxidation, and its sulphate should give double sulphates insoluble in the alkaline sulphates.

Further, peroxide of hydrogen separates an oxide, of which the atomic weight of the corresponding metal varies from 137.15 to 137.6; while the part not precipitated gives atomic weights varying from 137.85 to 139.9—variations of the same character as those obtained with the double sulphates (from 133.0 to 138.75) and with the acetate (from 135.1 to 140.7).—*Comptes Rendus*, cxxv., No. 20.

ON THE CHLORONITRIDES OF PHOSPHORUS.*

By H. N. STOKES.

(Concluded from p. 311).

Heptaphosphonitric Chloride, $P_7N_7Cl_{14}$.—After many distillations, this gave—

	Calculated for $P_7N_7Cl_{14}$.	Found.
P	26.75	26.57
N	12.11	12.12
Cl	61.14	61.28

Ratio, P : N : Cl = 1 : 1.01 : 2.02.

Molecular Weight. Solvent: Benzene.

Grms. solvent.	Grms. substance.	Elevation.	Molecular weight.	Percentage variation from theoretical.
47.09	3.5621	0.250°	808	-0.5
"	7.3979	0.523°	802	-1.2

Calculated for $P_7N_7Cl_{14}$, 811.7.

Heptaphosphonitric chloride is a nearly colourless rather viscous liquid, which does not solidify at -18° , and which boils at $289-294^\circ$ (corr.) at 13 m.m., undergoing some polymerisation. It is readily miscible with benzene, gasoline, and ether, and towards water shows the stability manifested by the preceding chloronitrides.

Residual Oily Phosphonitric Chlorides.†—The residue

which did not distil at 13 m.m. when the temperature of the bath was 370° , was boiled with water to remove the solid polyphosphonitric chloride, filtered, and carefully dried *in vacuo* over sulphuric acid. It is a thick liquid which gave on analysis:—

	Calculated for $(PNCl_2)_x$.	Found.
P	26.75	26.79
N	12.11	12.39
Cl	61.14	62.00

Ratio, P : N : Cl = 1 : 1.02 : 2.02.

Although the oil is doubtless a mixture, the above figures show that the constituents are members of the series $(PNCl_2)_x$. A determination of the mean molecular weight was made, with the following results:—

Grms. solvent.	Grms. substance.	Elevation.	Molecular weight.
46.72	4.130	0.178°	1326
46.72	7.853	0.348°	1290

Mean 1308. This does not lie far from that required by the formula $P_{11}N_{11}Cl_{22}$ (calculated, 1276). This result is interesting in as far as it shows that a phosphonitric chloride of this molecular weight may exist, that it is stable and miscible with benzene, gasoline, and ether, and that the molecular weight of the solid polymer described below, which is insoluble, is probably very much higher. The oil has a reddish brown colour, due to dissolved impurities, which are destroyed by heating with strong nitric acid. It cannot be distilled even at 13 m.m., as it polymerises almost instantly. In its behaviour towards water it resembles the preceding members of the series.

Polyphosphonitric Chloride, $(PNCl_2)_x$. This remarkable body, frequently alluded to above, is formed when any of the lower members are heated; slowly at 250° , and very rapidly at 350° . As the change is reversible, complete transformation cannot be effected, but reaches perhaps 90 per cent, the remainder consisting not only of the original phosphonitric chloride, but of others. These can be extracted by anhydrous benzene. The sample, the analysis of which is given, was prepared by heating pure triphosphonitric chloride in a sealed tube at $350-360^\circ$. The transparent elastic product was repeatedly extracted with benzene, dried over sodium, and the absorbed benzene removed in a vacuum with constant exhaustion over paraffin, and finally by heating *in vacuo* at 110° . Analysis gave—

	Calculated for $(PNCl_2)_x$.	Found.
P	26.75	26.78
N	12.11	12.27
Cl	61.14	60.45

Ratio, P : N : Cl = 1 : 1.01 : 1.08.

Polyphosphonitric chloride, when perfectly pure, is colourless and transparent, but is generally somewhat discoloured by traces of organic matter. Its most striking property is its elasticity. It may be drawn out like rubber, and shows even a greater tendency to rebound from hard surfaces. It is readily cut with the shears. It is insoluble in all neutral solvents, but absorbs benzene, swelling to many times its original volume and forming a jelly of but little coherence; on evaporating the benzene it returns to its original condition; ether is absorbed, but less readily, and other chloronitrides are taken up in a similar manner. Hot water slowly dissolves it with decomposition; in warm dilute ammonia it swells, gelatinises, and finally dissolves; hot caustic soda does not dissolve it readily, apparently insoluble sodium salts being formed. It begins to depolymerise towards 350° , and this change is rapid just below red heat, and is accompanied by partial fusion, the products being, as described above, a mixture of lower phosphonitric chlorides. These transformations are not modified by heating in an atmosphere of hydro-

* Published by permission of the Director of the United States Geological Survey. From the *American Chemical Journal*, vol. xix., No. 9, November, 1897.

† It is unlikely that these consist entirely of the original depolymerisation products; a portion is doubtless formed by polymerisation during distillation.

chloric acid. When perfectly pure, it leaves no residue whatever on distilling. No difference could be detected in the product formed from different phosphonitrilic chlorides.

Nitrilo-hexaphosphonitrilic Chloride, $P_6N_7Cl_9$.—The separation of this chloronitride, which does not belong to the phosphonitrilic chloride series, is described above. After four crystallisations from benzene, it gave—

		Calculated for $P_6N_7Cl_9$.	Found.
P		30.84	30.74
N		16.29	16.29
Cl		52.87	53.01

A molecular weight determination, kindly made for me by Dr. H. C. Jones, of the Johns Hopkins University, with a limited quantity of material, in benzene solution, gave 667; calculated for $P_6N_7Cl_9$, 603.5. This, with the analysis, suffices to establish the above molecular formula.

Nitrilo-hexaphosphonitrilic chloride strikingly resembles the phosphonitrilic chlorides. It fuses at 237.5° (corr.) and boils at $251-261^\circ$ (corr.) at 13 m.m. without change. The boiling-point coincides closely with that of hexaphosphonitrilic chloride ($261-263^\circ$ corr. at 13 m.m.), hence it is found associated with the latter. Heated in small quantities on foil, it volatilises without residue, but at a higher temperature in a sealed tube it undergoes a change the exact nature of which has not been determined, but which involves the formation of a substance resembling polyphosphonitrilic chloride, which yields lower phosphonitrilic chlorides on distilling. It crystallises in transparent prisms of not more than 1 m.m. in length, apparently rhombic, which are often united to acicular forms. When pulverised it becomes electrified. It dissolves in about 20 parts cold and 5 parts boiling benzene (approximate only), is more soluble in carbon disulphide, but less soluble in gasoline and in ether. Towards water it is nearly as stable as hexaphosphonitrilic chloride, but is slowly attacked when exposed to atmospheric moisture. Hot dilute ammonia dissolves it very slowly, but more rapidly when alcohol is added.

This body is obviously a secondary product of the reaction of phosphorus pentachloride and ammonium chloride, as it is never found when a pure phosphonitrilic chloride is polymerised and depolymerised. It is noteworthy that no indication of other bodies of a similar nature has been observed, although no reason appears why they should not be formed at the same time. Whether it is in reality hexaphosphonitrilic chloride in which three chlorine atoms are replaced by one of nitrogen or not, cannot be decided at present.

Examination of Auriferous Ores.—The gold present in such ores can be rapidly and safely determined by the following method, which is applicable to all kinds of ores:—75 grms. of borax (anhydrous), the same weight of crude tartar, and 50 grms. of red lead are mixed; 250 grms. of the pulverised ore are then added, along with 100 grms. of potash. This mixture is placed in a J crucible, and above it is spread a stratum of 75 grms. soda, and over this again a layer of sodium chloride 1.5 c.m. in thickness; lastly, 50 grms. of red lead. The crucible is placed in a gas-furnace, cautiously heated, at last strongly enough to bring it to the state of quiet flux. The melted mass is poured into a conical form, at the bottom of which it arrives as a lead regulus, which after the treatment is cupelled in a muffle furnace. If the ore contains no silver, a little silver free from gold is added. The globule of silver—possibly auriferous—is treated with nitric acid. The silver dissolves, and the residual gold is weighed. —*Chem. Zeitung and Swed. Teknisk. Tid.*

REGULATIONS

OF THE

THIRD INTERNATIONAL CONGRESS OF
APPLIED CHEMISTRY IN VIENNA, 1898.

1. THE Third International Congress of Applied Chemistry will be held in July, 1898, in Vienna, and will last about five days.

2. The subjects of the Congress are as follows:—

Consultations concerning important questions in all departments of Applied Chemistry, and particularly of those the solution of which is a matter of public interest. Agreement upon methods to be considered internationally valid for the analysis of such products as are valued upon the basis of their chemical composition.

Agreement upon methods for the use of the different chemical industries, to be considered internationally valid.

Discussion on questions of instruction in Applied Chemistry, and consultations upon general affairs of Chemistry; and commencement of a friendly understanding between the representatives of the different departments of Applied Chemistry at home and abroad.

3. For the settlement of the tasks of the Congress there will be two general meetings and a greater number of special consultations. Besides these there will be excursions for the purpose of visiting scientific institutions and establishments.

4. The special consultations of the Congress will be divided into twelve Sections.

Section I. General Analytical Chemistry and Chemical Instruments. (General methods of analysis; analytical apparatus, measuring instruments, hydrometers, &c.).

Section II. Chemistry of Food, Medical and Pharmaceutical Chemistry. (The chemical and physical analysis of foods, discussion of chemical questions regarding foods in trade which do not come under the head of another section; further questions of medical and pharmaceutical chemistry).

Section III. Agricultural Chemistry. (Agricultural chemistry; experiments and analysis of dairy products).

Section IV. Sugar Industry, Starch and Grape Sugar Manufacture.

1. Brewery and Malt Manufacture.

2. Alcohol and Yeast Industry.

Section V. Zymology.

Section VI. Ceno-Chemistry.

Section VII. Chemical Industry of Inorganic Substances. (Sulphuric acid, soda, and chloride of lime manufacture; alkaline industry; artificial manure production; lime and cement industry; industry of illuminants; glass, china, and earthenware manufacture).

Section VIII. Metallurgy and Industry of Explosives.

Section IX. Chemical Industry of Organic Substances. (Industry of aniline dyes; dyeing and printing; manufacture of pharmaceutical preparations; chemistry of fats, oils, and lubricants; paper and xylonite industry; tannery and glue manufacture).

Section X. Chemistry of the Graphic Industry. (Photochemistry, photographic and chemical printing, colour printing, &c.).

Section XI. Questions of instructions and general affairs of chemists.

Section XII. Electro-chemistry.

Questions which belong to several departments at once will be discussed in common meetings of the Sections concerned.

5. Anyone can become member of the Congress who is concerned with the theory or practice of chemistry or practically employed in any department of chemistry, as well as such persons and corporations as are interested in an undertaking in which chemical processes are applied, and also all those persons and societies which take an interest in the progress of Applied Chemistry.

6. Every member has to pay a fee of 10 florins, for

which he receives a members' card, which admits him to the general meetings and special sessions and all other Congress arrangements and entitles him to all publications of the Congress. Members who subscribe at least 100 florins for the benefit of the Congress will be mentioned specially in the Congress publications as Patrons of the Third International Congress for Applied Chemistry, Vienna, 1898.

7. Any surplus of the subscriptions will be devoted to benevolent purposes, and this will be decided at the second general meeting of the Congress.

8. Every member must enter his name at the beginning of the Congress in the lists of those Sections in which he will take part, and at the same time give his address during the Congress.

9. Every member of the Congress has a right to take part in the debates of the special consultations, and has the right of voting in the general meetings and the consultations of those Sections of which he is a member.

10. The languages to be spoken at the Congress are English, French, and German.

11. The business of the Congress will begin at the first general meeting. This will be opened by the President of the organising committee, and the record will be kept by the Council of the organising committee.

This meeting elects the Honorary President and the Honorary Vice-President, and nominates the President, the Vice-Presidents, the General Secretary, and Secretaries of the Congress.

12. The second general meeting will take place at the end of the Congress. This will be opened and presided over by the President of the Congress. The record will be kept by the General Secretary and the Secretaries of the Congress. In this an account will be given of the particulars of the Congress by the General Secretary, and the meeting will decide place and time of the Fourth International Congress of Applied Chemistry.

13. The first sitting of each branch section or sub-section will be opened by the President nominated by the organising committee, and the formation of the council of the special section or sub-section will be introduced by him. For the formation of the latter a President and a corresponding number of Vice-Presidents must be elected for the first sitting, and once for all a chief Secretary and several secondary Secretaries (according to the length of the discussions) for all the sittings of the section or sub-section. At the end of each sitting the President and the Vice-President for the following sitting are to be elected.

The Presidents, Vice-Presidents, and Secretaries elected by the organising committee remain at the same time members of the council of the special section or sub-section during all the sittings of the same.

14. The order of the day and the succession of the subjects of the consultation will be arranged independently by each section and sub-section. Reports which are printed and distributed to the members beforehand are generally not read aloud, and the debate upon these will be commenced at once by the President. Verbal reports should not last longer than twenty minutes. In the debate a speaker may not speak longer than ten minutes, and it is not permitted that he should speak on the same subject more than twice. The voting will be based on the members' lists of the special section or sub-section, and the simple majority decides. In case of equal votes the President gives the casting vote.

15. For the purpose of drawing up the Congress Record those reporters whose reports were not printed, as well as all speakers in the debate, must give a short account in writing of their speech to the first Secretary of the special section or sub-section not later than half an hour after the close of the meeting in question. The first Secretary must draw up the Record by means of the accounts received, and give it up if possible on the same day, together with the list of those present, to the General Secretary of the Congress.

16. The addresses to be given during the Congress, and

the reports to be presented, must be announced, not later than 30th of April, 1898, to the Special President of the section, or direct to the organising committee. Later announcements can only be accepted by the decision of the branch section or sub-section of the Congress concerned.

Lectures or reports which have to be printed must be sent at the latest on the 15th of April, 1898, to the General Secretary of the organising committee or to the President of the section concerned. The reports must not take more than five 8vo pages in print. The organising committee will decide regarding manuscripts of greater length of purpose for printing.

17. All conclusions or resolutions of the Congress will be laid before the respective Governments by the organising committee.

18. After the conclusion of the Congress a printed report will be given, free of charge, to all members.

19. About all other matters concerning the Congress not mentioned in the Congress Regulations the organising committee will give every information.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Extra Meeting, December 15th, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

PROFESSOR F. R. JAPP, LL.D., F.R.S., delivered the
Kekulé Memorial Lecture.

FRIEDRICH AUGUST KEKULÉ was born at Darmstadt on September 7th, 1829. Originally intended for the profession of an architect, he was induced, by hearing Liebig's lectures, to devote himself to chemistry. After studying under Liebig, he spent a year in Paris, where he became intimate with Gerhardt. Later on he resided for a time in London, making the acquaintance of Williamson and Odling. He always acknowledged the influence which these three chemists had exercised on the formation of his opinions. Kekulé's theories are based on Gerhardt's type theory; on Williamson's theory of polyvalent radicles, which, by their power of linking together other radicles, render possible the existence of multiple types; and on Odling's theory of mixed types, which was a deduction from Williamson's theory. Less consciously perhaps his opinions were influenced by E. Frankland's theory of the valency of elementary atoms, and by Kolbe's speculations on the constitution of organic compounds. Kekulé gathered together the various ideas which he found scattered throughout the writings of his predecessors, added to them, and welded the whole into the consistent system which forms our present theory of chemical structure. In 1857, in the course of a memoir on the constitution of fulminic acid, he gave a tabular arrangement of compounds formulated on the type of marsh gas, this being the earliest statement, although put forward only in an imperfect form, of the tetravalency of carbon. In the same year he published an important theoretical paper: "On the so-called Conjugated Compounds and the Theory of Polyatomic Radicles, which contains a complete system of multiple types and mixed types. In 1858 the celebrated paper, "On the Constitution and Metamorphoses of Chemical Compounds, and on the Chemical Nature of Carbon," appeared; it embodies the fully-developed doctrine of the tetravalency of carbon, together with Kekulé's views on the linking of atoms and on the valency of such chains of atoms—the foundation on which our modern system of constitutional formulæ rests. In 1865 Kekulé put forward his well-known benzene theory—the crowning achievement, in his hands, of the doctrine of the linking of atoms, and the

most brilliant piece of scientific prediction to be found in the whole range of organic chemistry. The conception of closed chains, or cycloids, which he thus introduced, has shown itself to be capable of boundless expansion.

Kekulé published the first instalment of his "Lehrbuch der Organischen Chemie" in 1859. The work was never finished, but it was instrumental in widely disseminating Kekulé's views, and exercised enormous influence on the development of the science.

Kekulé obtained the *venia legendi* in Chemistry at the University of Heidelberg in 1856. Two years later he was called, as ordinary professor, to the University of Ghent, where he remained until 1867, when he was appointed to the Professorship of Chemistry in the University of Bonn, a post which he continued to hold until his death on July 13, 1896. During his later years he suffered from bad health.

The characteristic note of Kekulé's great theoretical creation, the chemistry of structure, is the treatment of the problem of isomerism—the problem which first necessitated the use of constitutional formulæ—as one of geometrical symmetry. Kekulé's formulæ, freed from the fetters of the type theory with which he had first encumbered them, were merely more or less symmetrical geometrical figures. In order to predict the number of substitution compounds, it was only necessary to consider the degree of dissymmetry of the parent compound: the less the symmetry, the greater the number of isomeric substitution compounds. The extraordinary fertility of this conception is shown by the development which it has undergone at the hands of van't Hoff, J. Wislicenus, von Baeyer, and others.

The accuracy of Kekulé's predictions has done more to inspire a belief in the utility of legitimate hypotheses in chemistry, and has therefore done more for the deductive side of the science, than that of almost any other investigator. His work stands pre-eminent as an example of the power of ideas. A benzene formula, consisting of a few chemical symbols jotted down on paper and joined together by lines, has supplied work and inspiration for scientific organic chemists during an entire generation, and affords guidance to the most complex industry the world has yet seen.

Dr. HUGO MÜLLER, as probably the oldest personal friend of Kekulé present, moved a cordial vote of thanks to Professor Japp for his eloquent lecture, and added his special appreciation of the admirable and exhaustive manner in which the lecturer had accomplished his task. A considerable effort was needed to realise the condition in which organic chemistry stood fifty years ago, in order to recognise the vast advances which have been made in the interval. It may be truly claimed for Kekulé that he holds a foremost position amongst those reformers who have initiated this progress. It was here, in London, that Kekulé first conceived the ideas which, in their further development, assumed the shape of his "chemistry of carbon" and "benzene theory," and, being in those days in almost daily intercourse with him, he well recollected the eagerness and enthusiasm with which the problems which occupied his mind were discussed.

Soon afterwards, in Heidelberg, and then in Ghent, his affable manner and sociability attracted a number of devoted pupils, who became active fellow workers, and thus his teaching bore fruit in all directions.

Unfortunately, not long after he had been settled in Bonn, his health gradually gave way, and he suffered much from nervous prostration and an irksome degree of deafness, which at times much depressed him. His power of work became greatly impaired, and notwithstanding repeated heroic efforts, even his Handbook had to remain unfinished.

Professor THORPE, in seconding the resolution, also desired to give expression to the sense of obligation which the Society was under to Professor Japp for the thoughtful and eminently impartial address which he had

given. There was, however, one slight but characteristic omission in the lecture. In enumerating Kekulé's students, Dr. Japp had neglected to make any reference to himself. It was, no doubt, that same feeling of piety to which he had borne witness in the course of his lecture on the part of another which induced Dr. Japp to comply so readily as he had done with the request of the Council that he should undertake the weighty and responsible duty of delivering this address. His personal intercourse, as a student, with the master had, we may take it, quickened his appreciation of his work. At the same time, as would be evident, it had in no sense diminished his critical faculty. The audience had recognised that the lecture was a truthful and well-balanced account, written impartially and in the true spirit of history, of the origin and fruit, so far as this had been gathered, of the great chemist's labours.

Some reference had been made to the fact that he (the speaker) had enjoyed the good fortune of also being a student under Kekulé, and of being associated with him, in some small degree, in certain experimental work which he undertook during the first years of his professorship in the magnificent institution which Germany owes to Hofmann. It is a curious coincidence that Hofmann, like Kekulé, might have become an architect if destiny, as in Kekulé's case, had not intended that he should be a chemist. During the late sixties there was no sign of the decay in intellectual vigour which a few years later became so sadly obvious. At that time the great generalisation which we associate with Kekulé's name was still, to some extent, on its trial, and it had to withstand the assaults which were from time to time delivered by keen and active opponents in other schools of chemical thought. It happened that very shortly after the speaker's entrance into Kekulé's laboratory he was called upon to handle the weapons which Kekulé himself placed in his hands in order to defend a small but apparently vulnerable point in the theory. That circumstance proved of incalculable benefit to him, in that it brought him into intimate personal contact with Kekulé, and enabled him to see something of his methods of work, and of the springs of his intellectual activity. Dr. Japp has ably testified to Kekulé's merits as a teacher. Kekulé, indeed, was one of the very best expositors, with the single possible exception of Kirchhoff, to whom it had been the speaker's lot to listen. As a laboratory teacher he was excellent. He was a most severe judge of work, striving to exact the same high manipulative finish, the same neatness and order, which he invariably bestowed on everything he did, and he was absolutely intolerant of anything slovenly or "sloppy." But it was as a lecturer that he was seen at his best. He was singularly luminous as a thinker, a close and accurate reasoner, with a remarkable power of concentrated expression. He was not a rapid speaker, and he never indulged in those rhetorical flights with which Hofmann occasionally was wont to electrify an audience. His language was apt and well chosen, and his delivery easy and natural. His lecture-table was never overburdened with "experiments"; those he showed were strictly proper to the subject in hand. To see him handle the chalk was in itself a liberal education. Although everything appeared to be so easy and natural, an attentive critic could hardly fail to perceive that the lecture had been carefully thought out beforehand, possibly over a longer period of time than it took to utter. Every detail would seem to have been considered, even to the particular places on the blackboard where the formulæ should appear.

During the later period of his life, Kekulé, unfortunately for Science, was comparatively sterile. Those who knew him, however, would be the first to affirm that this seeming apathy sprang from no natural indolence. There is no doubt that he suffered, even in the early period of middle life, from the intense stress and strain of his mental labours prior to the Ghent period. He had too surely exemplified the sad truth of Liebig's

saying, to which Dr. Japp had referred, that he who would become a great chemist must pay for his pre-eminence by the sacrifice of his health. There is reason to know that it was the consciousness of failing power which prevented him from finishing much to which he had put his hand, and that his fastidiousness and his sense of "finish," amounting almost to hypercriticism, restrained him from publishing what he realised fell short of his ideal. What he has left us, however, is an imperishable monument to his genius.

The President said that there was little to add to Professor Japp's exhaustive eulogy of the life and work of Kekulé. His own early relations, however, with the great chemist whose life work the Society was commemorating might have some interest to the members and ought to be told. While a student with Lord Playfair at Edinburgh, in the session 1866-67, he made his first contribution to Science in the shape of a little paper entitled, "On the Oxidation of Phenyl Alcohol and a Mechanical Arrangement adapted to Illustrate Structure in the Non-saturated Hydrocarbons." This note appeared in the *Proceedings of the Royal Society of Edinburgh*, and he was so desirous of becoming known to Kekulé as a student of his theory of the aromatic bodies that a specimen model was sent to Ghent. Lord Playfair addressed a letter to Kekulé stating that he (Professor Dewar) was very anxious to work in his laboratory. The reply was, "Come," and the reception and kindness he received from Kekulé has always had his profound gratitude.

The summer of 1867 was thus spent in the private laboratory of Kekulé. Before leaving Edinburgh, he had been working on the coal tar bases, and a supply was taken with him to Ghent. There he began the study of the oxidation products of picoline, and at the British Association Meeting at Norwich, in 1868, an account of the separation of dicarbopyridinic acid, the analogue of phthalic acid in the benzene series, was given. At the same meeting, he gave a paper on "Kekulé's Model to Illustrate Graphic Formulæ." This is the succinct history of the beginning of the pyridine-benzene analogy. His old friend Koerner had speculated in the same direction, and he (Prof. Dewar) might confess that in his opinion they both had received too much credit for an extension of the benzene theory to pyridine. At a distance of thirty years, to look back and call to mind the presence and personality of the great chemist as he knew him was indeed a pleasure. He was a man of noble mien, handsome, dignified, and yet of a homely and kindly disposition. He was a severe critic, having a haughty contempt for the accidental and *bizarres* in scientific work. His originality and suggestiveness seemed endless, so that he had no need to commit scientific trespass or to follow just in the wake of other people's ideas. Everything that passed through the Kekulé alembic was indeed transmuted into pure gold. His precision of thought and diction rendered his papers profoundly suggestive to other workers. His great work will always live in the history of our Science, and his loving memory will be for ever enshrined in the hearts of his pupils.

CORRESPONDENCE.

THE EXPLOSION AT DARTFORD.

To the Editor of the Chemical News.

SIR,—Having received numerous enquiries in respect to the fatal explosion which occurred at our Dartford works on Wednesday, the 15th inst., and so many erroneous versions having appeared in the public press, we think it well to state the simple facts of the case.

The deceased, Mr. Lewis Jones (qualified chemist), was

mixing a preparation of erythrol tetranitrate—a remedy which is now somewhat extensively prescribed by the medical profession in cardiac affections. The process consisted in diluting erythrol tetranitrate with finely-powdered lactose by gently stirring the substances in a mortar; no pounding or grinding was required. The quantity of erythrol in the possession of the deceased was four ounces. Our process for dealing with this substance was adopted after a series of careful experiments, and it has always been performed by competent chemists who knew the dangers of such nitrous compounds. The process has been carried out by us many times during the past eighteen months without the slightest mishap. It has been the rule of the chief of the department to caution those who handled it; and the deceased received such a warning. The erythrol tetranitrate was kept under lock and key in a dark closet. The cause of the explosion we can only attribute to some extraordinary accident; unfortunately there was no witness to the actual carrying out of the operation by the deceased on this occasion. The force of the explosion was violent, but local.

We need hardly add that we are deeply pained by this sad occurrence, which has resulted in the death of an employé who had gained our high esteem.—We are, &c.,

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CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 20, November 15, 1897.

Reaction of Hydrogen upon Sulphuric Acid.—M. Berthelot.—The author finds that there is an important reaction between hydrogen and sulphuric acid. 0.5 gm. of H_2SO_4 (boiled) was sealed in a glass tube with 11 c.c. of hydrogen; at the end of six hours, at a temperature of 250°, all the hydrogen was absorbed, with the production of water and sulphurous acid. The reaction takes place also at the ordinary temperature, but it is much slower; it does not occur with dilute acid, and light has no appreciable effect.

Influence of Oxygen upon the Decomposition of the Hydracids by Metals, and especially by Mercury.—M. Berthelot.—Mercury is generally considered to have no particular action on gases with which it may be in contact, with the exception of a few such as chlorine, nitrous acid, &c.; but there are gases which have a slow action, among which are HCl, under certain conditions of temperature and time and in the presence of free oxygen.

Direct Reaction of Sulphuric Acid with Mercury at the Ordinary Temperatures.—M. Berthelot.—Boiled sulphuric acid has a slow action on mercury, forming a sulphate, and giving off sulphurous acid when carbonic acid is passed through it. The reactions described in this paper only take place when the sulphuric acid is at the maximum of concentration.

Influence of Surfusion upon the Congelation-point of the Solutions of Potassium Chloride and Sugar.—F. Raoult.—Previous experiments have shown that the value of K in the equation $C = C'(1 - KS)$ for aqueous solutions of chloride of sodium and alcohol is not constant, as was generally admitted, but that it varies sensibly with the concentration. Further experiments show that this is not the case with aqueous solutions of chloride of potassium and of cane-sugar. The new experiments were carried out with the same apparatus as the former ones and the table of figures shows that the molecular lowering

of the congelation-point varies in a very different manner with regard to the concentration of the substances mentioned.

Action of Water upon Phosphorous Trichloride and Phosphorous Oxichloride.—A. Besson.—The reaction between trichloride of phosphorus and water in excess gives phosphorous acid and hydrochloric acid; but when the trichloride is in excess, several peculiarities are noticed. No matter how we arrange for the reaction between a small quantity of water and PCl_3 , we always find a small quantity of a phosphorous oxichloride resulting from the incomplete reaction $\text{PCl}_3 + \text{H}_2\text{O} = 2\text{HCl} + \text{POCl}$. This latter is a solid body of the consistence of paraffin, with a smell similar to that of phosphoric oxichloride or chloride of phosphoryl, POCl_2 . This body is insoluble in the usual solvents, and is analogous in the phosphoric series to NOCl , AsOCl , &c.

Production of Strontium Sulphide by means of Sulphuretted Hydrogen and Strontia or Carbonate of Strontium. Influence of Temperature.—J. R. Mourelle.—It was found that temperature had a curious influence on the results in the carrying out of this process. A porcelain tube containing either strontia or carbonate of strontia is placed in a furnace, and sulphuretted hydrogen gas is passed over it, at first in the cold. The tube is then gradually warmed to a bright red heat, taking care that the current of gas is strong enough to carry off the water formed by the reaction. When the transformation is complete, a slow current of dry hydrogen is substituted for the sulphuretted hydrogen; after which the tube is allowed to cool, and the contents, monosulphide of strontium, may be withdrawn; this sulphide is not phosphorescent. At a red heat the formula of the reaction is $2\text{SH}_2 + \text{SrO} = \text{SrS} + \text{H}_2\text{O}$. If the temperature is not sufficiently high, the water condenses in the tube and attacks the sulphide already formed, and a mass has been found containing 22 per cent of strontic hydrate and smelling strongly of sulphuretted hydrogen.

Production of Volatile Fatty Acids from the Washing Waters of Wools.—A. and P. Buisine.—The authors think that the production of these volatile acids is now of commercial importance, and they give a *résumé* of the method of procedure by which they are distilled, entangled with and condensed by watery vapour. The waters are first allowed to ferment for eight days, then boiled to drive off the ammonia, then acidulated with sulphuric acid and distilled. The volatile fatty acids found in the largest quantities are acetic and propionic, but we also get a fair percentage of butyric, valerianic, caprylic, and benzoic acids, with traces of formic and caprylic acids and phenol. Amongst other applications, this mixture of raw fatty acids is particularly suitable for the production of acetone, methyl-ethylacetone, and the higher acetones which are comprised by the mixture known as "oil of acetone."

Decomposition of Chloroform, Bromoform, and Chloral by a Solution of Potash.—A. Desgrez.—Bright sunlight has an accelerating, and darkness a retarding, influence on the decomposition of chloroform by this means, but potash without the mediation of water has no effect. Chloral has the same action, but it occurs more rapidly.

NOTES AND QUERIES.

* Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Oxidation of Organic Matter.—I have endeavoured to find if any records exist of observations on the oxidation, by atmospheric oxygen, of organic matter in commercial use, e.g., of rope, wood, gums, caoutchouc, &c. The conditions which favour such action appear to be very different from those in the cases of metals. If any correspondent can give any information they will oblige.—H. V.

Destruction of Moths by Formic Aldehyd.—Formic aldehyd is so powerful a disinfectant, and so destructive to most low forms of life, that I think it will help us to solve the great "moth" problem of the household. I propose to pack up furs and winter coats as soon as they are done with in the spring in a large tin box containing vapour of formic aldehyd; but before doing so I should like some of your correspondents who may be better acquainted with the properties of formic aldehyd than I am to tell me—(1) Will it have any injurious action on the fur? (2) Will it prevent the eggs hatching? (3) Will it kill the maggots when hatched? (4) Will it kill the moths or prevent them from laying eggs? I am sure many readers of the CHEMICAL NEWS will find replies to these questions very useful.—MOTR-EATEN.

MEETINGS FOR THE WEEK.

MONDAY, 3rd.—Society of Chemical Industry, 8. "Standard Methods of Tanning Analysis as adopted by the International Association of Leather Trades Chemists, with remarks thereon," by Prof. H. R. Procter and Dr. J. G. Parker. "Extraction of Tanning Materials at various Temperatures," by Dr. J. G. Parker. "Neatsfoot Oil," by J. H. Coste, F.I.C., and E. J. Parry, B.Sc., F.I.C.

TUESDAY, JANUARY 4th. } Royal Institution, 3. (Christmas Lec-
THURSDAY, JANUARY 6th. } tures). "Principles of the Electric
SATURDAY, JANUARY 8th. } Telegraph," by Prof. Oliver Lodge.

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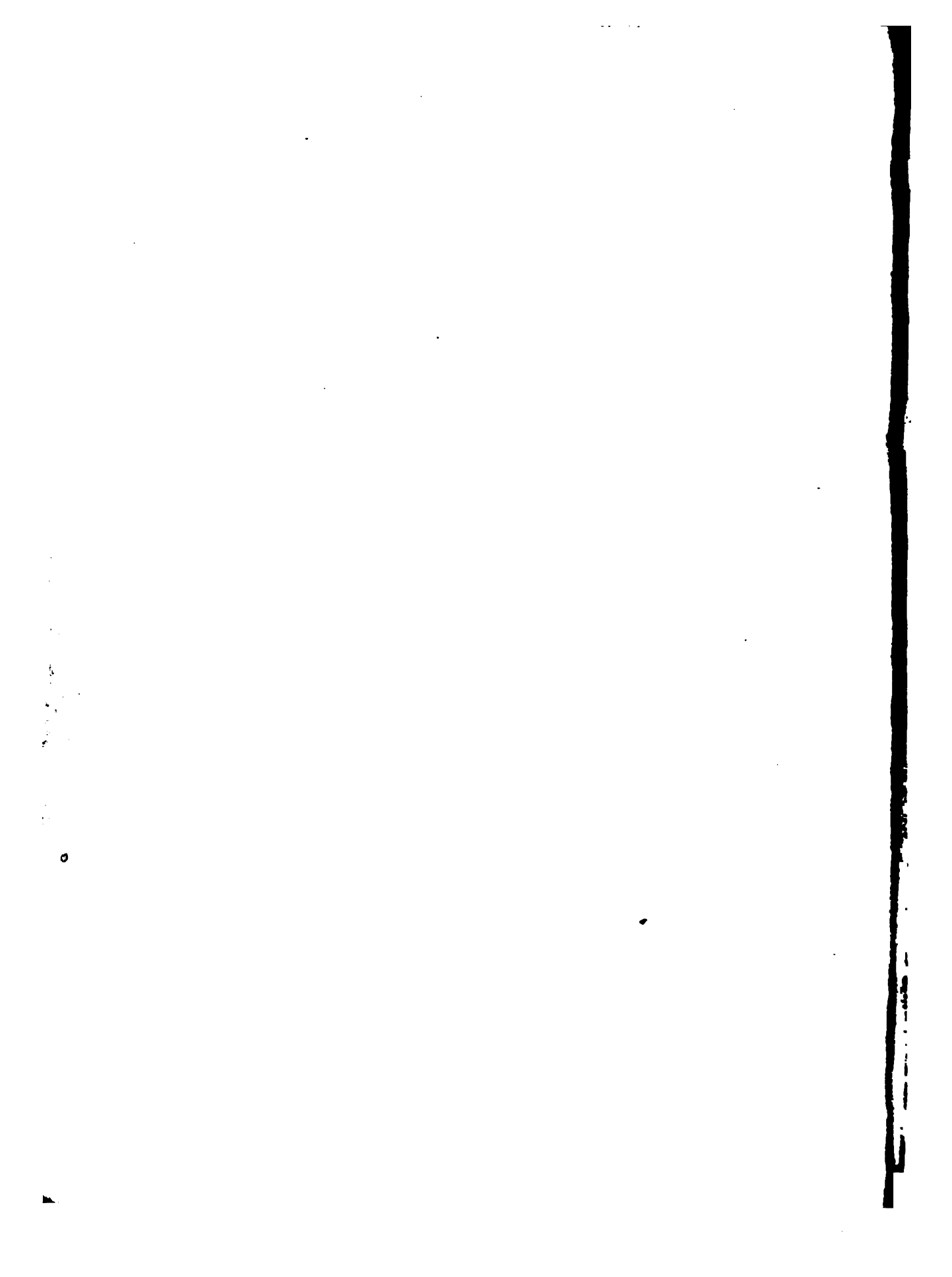
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